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FINAL

ADMINISTRATIVE RECORD

CSF Negative Exposure Assessment Report - May

Libby, Montana Asbestos Project

Sample Processing

July 30, 2003

Contract No. DTRS57-99-D-00017
Task Order No. 20

Prepared for:

U.S. Department of Transportation
Research and Special Programs Administration
John A. Volpe National Transportation Systems Center
Environmental Engineering Division, DTS-33
55 Broadway, Kendall Square
Cambridge, Massachusetts 02142

Prepared by:

CDM Federal Programs Corporation
One Cambridge Place
50 Hampshire Street
Cambridge, Massachusetts 02139

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Prepared by: _____ Date: 7/30/03
Jeff Montera
CSF Task Manager

Reviewed by: _____ Date: 7/30/03
Doug Updike
Site Health and Safety Officer

Reviewed by: _____ Date: 7/30/03
George DeLullo
QA Manager

CDM

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Acronyms

ASTM	American Society for Testing and Materials
CDM	CDM Federal Programs Corporation
cm^2	centimeter squared
CSF	close support facility
EPA	U.S. Environmental Protection Agency
EXC	excursion
f/cc	fibers per cubic centimeter
f/mm^2	fibers per square millimeter
H&S	health and safety
HEPA	high efficiency particulate air
LA	Libby amphibole
L/min	liters per minute
OSHA	Occupational Safety and Health Administration
PCM	phase contrast microscopy
Reservoirs	Reservoirs Environmental Services, Inc.
s/cc	structures per cubic centimeter
s/cm^2	structures per square centimeter
SOP	standard operating procedure
TEM	transmission electron microscopy
TWA	time weighted average
Volpe	U.S. Department of Transportation Volpe Center
≥	greater than or equal to
≤	less than or equal to
µm	micron

Section 1
Introduction

A task-based negative exposure assessment was conducted by CSF personnel on May 21, May 23, May 27, and May 28, 2003 to determine potential exposures at the CDM Federal Programs Corporation (CDM) close support facility (CSF). The purpose of this report is to present the results of that assessment and the corrective actions taken.

1.1 Soil Sample Processing

CDM has been tasked by the U.S. Department of Transportation Volpe Center (Volpe) to prepare soil samples collected at the Libby site prior to their analysis. The preparation includes drying, sieving, splitting, and grinding. These procedures were developed to produce a sample with well-homogenized material of a standard particle size.

1.2 CDM Close Support Facility Location and Description

CDM prepares soil samples collected in Libby at its CSF located in Denver, Colorado. The CSF consists of approximately 3,000 square feet of space, which includes an office, drying room, wet chemistry room, storage/receiving room, equipment storage room, and the main laboratory. Both the ventilation hood and drying oven are vented to a high efficiency particulate air (HEPA) filter unit designed to remove 99% of particles 0.3 microns (μm) or greater. Figure 1-1 provides floor plan of the CSF.

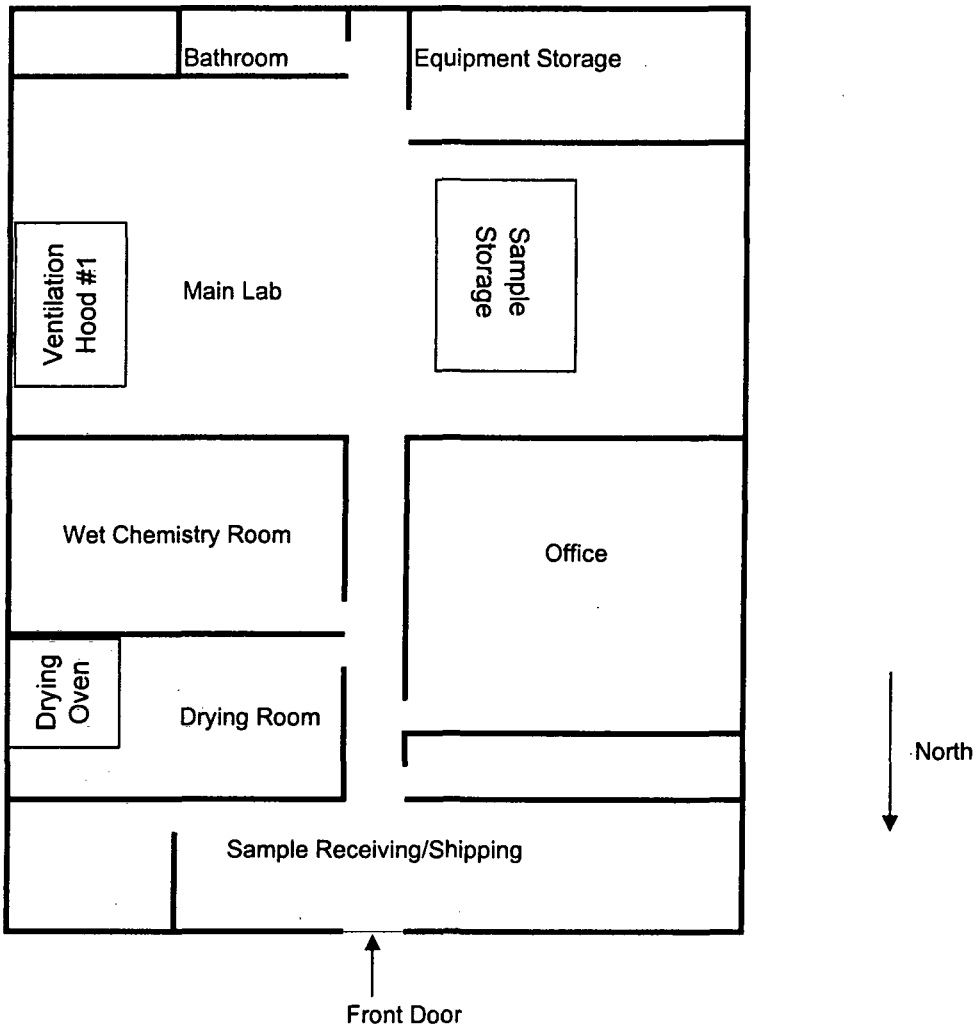
1.3 Close Support Facility Activities

The following is a list of activities that are performed by CDM personnel at the CSF:

- Sample receipt and check-in
- Sample storage
- Sample drying
- Archive sample splitting
- Sample sieving
- Fine sample grinding
- Fine sample splitting and archiving
- Sample packaging and shipping
- CSF measurements
- Documentation
- Equipment decontamination

All sample preparation procedures are described in the Close Support Facility Soil Preparation Plan (CDM 2003).

Figure 1-1 CSF Floor Plan



Section 2
Negative Exposure Assessment

In accordance with the soil preparation plan (CDM 2003), a task-based negative exposure assessment was conducted to assess potential exposures and to document facility cleanliness at the CSF. The assessment consisted of ambient air samples, personal air samples, and microvacuum dust samples. During the negative exposure assessment, CSF personnel were in modified level-C personal protective equipment. CDM's CSF personnel performed the sampling. Analysis was performed by Reservoirs Environmental Services, Inc. (Reservoirs).

2.1 Air Samples

2.1.1 Ambient Air Samples

Four ambient air samples were collected on May 27, 2003. Sampling locations included the main laboratory, office, drying room, and sample receiving room (Figure 2-1). Samples were collected using high-volume pumps at flow rates ranging between 8.35 and 8.58 liters per minute (L/min). Pumps were calibrated in accordance with section 7.2.3 of U.S. Environmental Protection Agency (EPA) standard operating procedure (SOP) 2015 *Asbestos Sampling* (EPA 1994) (Appendix B). Once calibrated, pumps were placed either on stands or on tables, with cassettes positioned downward and sampling was conducted in accordance with section 7.4.2 of EPA 2015.

2.1.2 Personal Air Samples

Personal air samples were collected by task (i.e., activity) to allow for differentiation among various exposure scenarios within the CSF. The two tasks sampled were sample receiving/sample coordinator and sample preparation. The sample receiving/sample coordinator position is responsible for checking in samples from the field, sorting samples in storage boxes, shipping samples and conducting general office activities. The sample preparation position is responsible for sieving, grinding and splitting samples. Each task was sampled for 3 consecutive processing days. During each day, a time-weighted average (TWA) and a 30-minute excursion sample were collected for each task. TWA samples were collected using personal air sampling pumps at flow rates ranging between 1.6 and 1.8 L/min. Thirty minute excursion samples were collected using personal air sampling pumps at flow rates ranging between 1.6 and 1.8 L/min.

2.1.3 Air Sample Analysis

All air samples collected were analyzed by two methods:

1. NIOSH 7400 – Asbestos and Other Fibers by phase contrast microscopy (PCM) (NIOSH 1994)

2. Appendix A to Subpart E of Part 763 - Interim Transmission Electron Microscopy Analytical Methods--Mandatory and Nonmandatory—and Mandatory Section to Determine Completion of Response Actions (also known as the AHERA method)

2.2 Dust Samples

2.2.1 Microvacuum Dust Samples

Four microvacuum dust samples were collected on May 28, 2003. Samples were collected from inside the ventilation hood, the floor beneath the ventilation hood in the main laboratory, the floor beneath the drying oven in the drying room, and the floor in the center of the office (Figure 2-1). Samples were collected using personal air sampling pumps at a volume of 2.15 L/min. A composite sample consisting of three independent 100 square centimeter (cm^2) grids were sampled for 2 minutes each for a total of 300 cm^2 and 6 minutes per sampling location (composite dust sample). All samples were collected in accordance with American Society for Testing and Materials (ASTM) Standard D 5755-95 "Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations (Appendix C)."

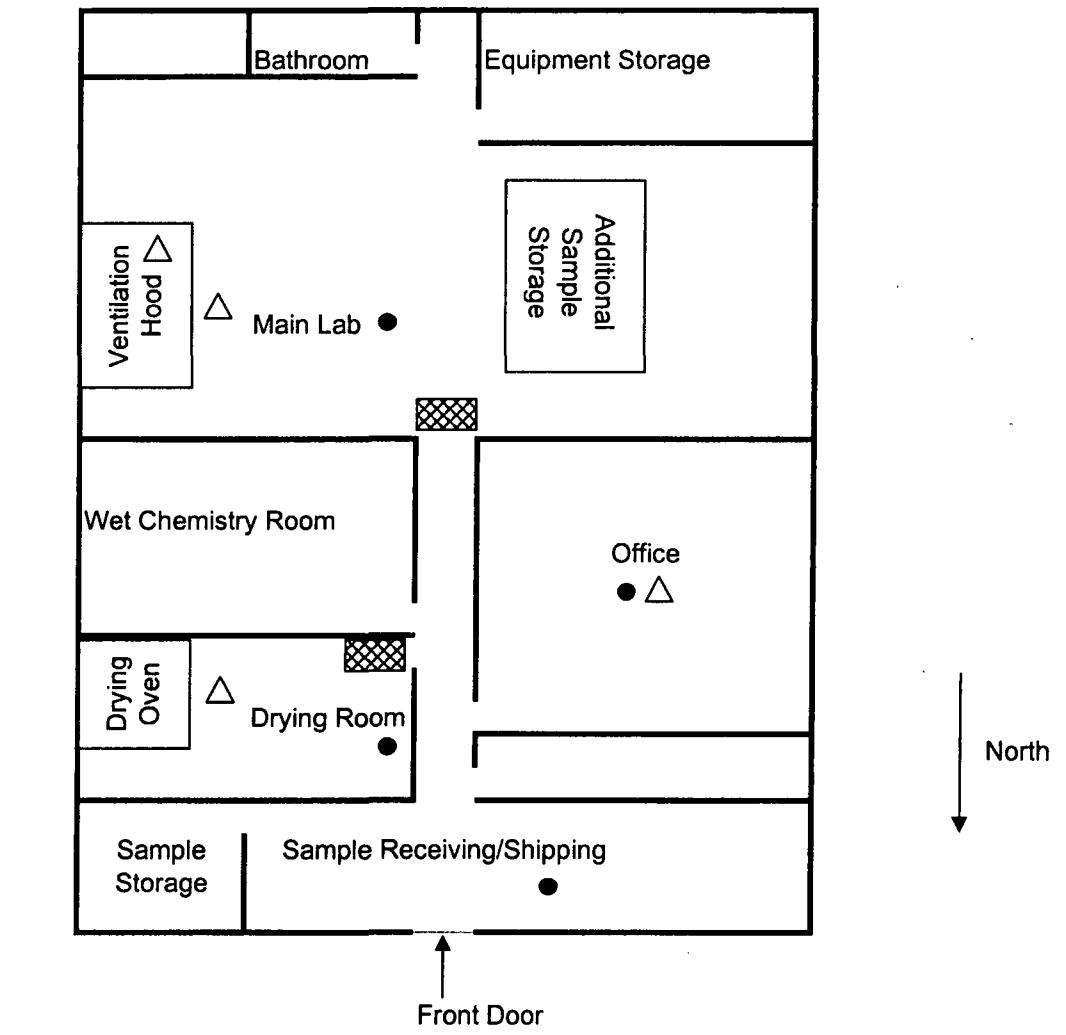
2.2.2 Dust Sample Analysis

All dust samples were analyzed using transmission electron microscopy (TEM) which stipulates the AHERA counting protocol (ASTM D5755-95).

2.3 Deviations

Due to staff illness and holidays the personal air samples were not completed in three consecutive days. It was, however, conducted in three consecutive processing days (May 21, May 23, and May 27). There were no other deviations to the collection, preparation, and analysis of the samples.

Figure 2-1 Negative Exposure Assessment Sampling Locations



Section 3
Results

A sample collection key is presented in Table 3-1. A summary of all PCM sample results is presented in Table 3-2, and a summary of the TEM sample results is presented in Table 3-3. Detailed bench sheets are included as Appendix A.

Table 3-1. Negative Exposure Assessment Sample Collection Key

	Index ID	Sample Type	Sample Date	Personnel/Area	Duration (min)	Volume (L)	Area (cm^2)
Personal Air Samples	CS-11339	8-hour TWA	5/27/03	Sample prep.	420	578.7	n/a
	CS-11340	30-min EXC	5/27/03	Sample prep.	30	48.9	n/a
	CS-11341	8-hour TWA	5/27/03	Sample coord.	375	607.5	n/a
	CS-11342	30-min EXC	5/27/03	Sample coord.	32	50.7	n/a
	CS-11343	8-hour TWA	5/28/03	Sample prep.	497	780.3	n/a
	CS-11344	30-min EXC	5/28/03	Sample prep.	30	53.4	n/a
	CS-11345	8-hour TWA	5/28/03	Sample coord.	472	731.9	n/a
	CS-11346	30-min EXC	5/28/03	Sample coord.	30	50.2	n/a
	CS-11347	8-hour TWA	5/29/03	Sample prep.	533	853.8	n/a
	CS-11348	30-min EXC	5/29/03	Sample prep.	32	49.6	n/a
	CS-11349	8-hour TWA	5/29/03	Sample coord.	488	807.8	n/a
	CS-11350	30-min EXC	5/29/03	Sample coord.	30	53.4	n/a
Ambient Air Samples	CS-11351	Ambient air	5/29/03	Main laboratory	520	4175	n/a
	CS-11352	Ambient air	5/29/03	Office	509	4275.6	n/a
	CS-11358	Ambient air	5/29/03	Soil drying	503	4187.5	n/a
	CS-11359	Ambient air	5/29/03	Receiving	491	4023	n/a
Dust Samples	CS-11360	Microvacuum	5/29/03	Inside hood	6*	n/a	300
	CS-11361	Microvacuum	5/29/03	Floor beneath hood	6*	n/a	300
	CS-11362	Microvacuum	5/29/03	Soil drying	6*	n/a	300
	CS-11363	Microvacuum	5/29/03	Office	6*	n/a	300

* Microvacuum samples were collected in three independent 100 cm^2 (2 min. each) areas for a total of 300 cm^2 (6 min. total).

cm^2 – square centimeter, EXC – excursion, L – liter, min = minute, n/a – not applicable, TWA – time weighted average

Table 3-2 Summary of the Negative Exposure Assessment PCM Results

	ID Number	Sample Type	Sample Date	Personnel/Area	Fiber Concentration* (f/cc)	TWA (f/cc)	30 Minute Excursion (f/cc)
Personal Air Samples	CS-11339	8-hour TWA	5/27/03	Sample prep.	0.011	0.018	n/a
	CS-11340	30-min EXC	5/27/03	Sample prep.	<0.055	n/a	<0.0055
	CS-11341	8-hour TWA	5/27/03	Sample coord.	0.011	0.009	n/a
	CS-11342	30-min EXC	5/27/03	Sample coord.	<0.053	n/a	<0.053
	CS-11343	8-hour TWA	5/28/03	Sample prep.	0.007	0.007	n/a
	CS-11344	30-min EXC	5/28/03	Sample prep.	<0.050	n/a	<0.050
	CS-11345	8-hour TWA	5/28/03	Sample coord.	0.009	0.009	n/a
	CS-11346	30-min EXC	5/28/03	Sample coord.	<0.054	n/a	<0.054
	CS-11347	8-hour TWA	5/29/03	Sample prep.	0.005	0.006	n/a
	CS-11348	30-min EXC	5/29/03	Sample prep.	<0.054	n/a	<0.054
	CS-11349	8-hour TWA	5/29/03	Sample coord.	0.005	0.005	n/a
	CS-11350	30-min EXC	5/29/03	Sample coord.	<0.050	n/a	<0.050
Ambient Air Samples	CS-11351	Ambient air	5/29/03	Main lab	0.001	n/a	n/a
	CS-11352	Ambient air	5/29/03	Office	0.001	n/a	n/a
	CS-11358	Ambient air	5/29/03	Soil drying	<0.001	n/a	n/a
	CS-11359	Ambient air	5/29/03	Receiving	0.001	n/a	n/a

TWA = time weighted average EXC = excursion f/cc = fibers per cubic centimeter n/a = not applicable

* Total asbestos fiber concentration

Table 3-3 Summary of the Negative Exposure Assessment TEM Results (Air)

	ID Number	Sample Type	Sample Date	Personnel/Area	Asbestos Concentration* (s/cc)	Asbestos Mineral
Personal Air Samples	CS-11339	8-hour TWA	5/27/03	Sample prep.	<0.006	ND
	CS-11340	30-min EXC	5/27/03	Sample prep.	<0.0716	ND
	CS-11341	8-hour TWA	5/27/03	Sample coord.	0.013	LA
	CS-11342	30-min EXC	5/27/03	Sample coord.	<0.0690	ND
	CS-11343	8-hour TWA	5/28/03	Sample prep.	0.013	LA
	CS-11344	30-min EXC	5/28/03	Sample prep.	<0.0655	ND
	CS-11345	8-hour TWA	5/28/03	Sample coord.	<0.0048	ND
	CS-11346	30-min EXC	5/28/03	Sample coord.	<0.0697	ND
	CS-11347	8-hour TWA	5/29/03	Sample prep.	<0.0046	ND
	CS-11348	30-min EXC	5/29/03	Sample prep.	<0.0706	ND
	CS-11349	8-hour TWA	5/29/03	Sample coord.	<0.0043	ND
	CS-11350	30-min EXC	5/29/03	Sample coord.	<0.0655	ND
Ambient Air Samples	CS-11351	Ambient air	5/29/03	Main lab	<0.0021	ND
	CS-11352	Ambient air	5/29/03	Office	<0.0020	ND
	CS-11358	Ambient air	5/29/03	Soil drying	<0.0008	ND
	CS-11359	Ambient air	5/29/03	Receiving	0.013	C

ND = None Detected
Average Grid Opening = 0.0110 mm^2
s/cc – structures per cubic centimeter
C = Chrysotile
Filter Material = Mixed Cellulose Ester
Filter Diameter = 25 mm
Effective Filter Area = 385 mm^2
LA = Libby Amphibole
* Total asbestos fiber concentration

Table 3-4 Summary of the Negative Exposure Assessment TEM Results (Dust)

ID Number	Sample Type	Sample Date	Personnel/Area	Sample Area (cm^2)	Asbestos Concentration* (s/cm^2)
CS-11360	Microvacuum	5/29/03	Inside hood	300	3700
CS-11361	Microvacuum	5/29/03	Floor beneath hood	300	3000
CS-11362	Microvacuum	5/29/03	Soil drying	300	2900
CS-11363	Microvacuum	5/29/03	Office	300	7900

s/cm^2 – structures per square centimeter

* Total asbestos concentration

Section 4
Findings and Corrective Actions

4.1 Findings

Results from the PCM analysis were received from Reservoirs on June 2, 2003. TEM results were also received from Reservoirs June 12, 2003.

4.1.1 PCM

Ambient air samples and personal air samples were analyzed by PCM. The evaluation criteria, corrective action, and results for each are presented below. All evaluation criteria and corrective actions outlined in this section are described in the Close Support Facility Soil Preparation Plan (CDM 2003).

Ambient

Evaluation criteria: Ambient air samples are considered acceptable if the PCM result is less than or equal to (≤) 0.01 fibers per cubic centimeter (f/cc).

Corrective action: If the evaluation criterion is not met, then the CSF will be re-cleaned by wet-wiping and vacuuming the affected area and then ambient air samples will be recollected.

Results: No ambient air PCM results were above the evaluation criteria. Therefore, no corrective action was taken based on these results.

Personal Sample Results

Evaluation criteria: Time weighted average personal air samples are considered acceptable if the PCM result is ≤ 50 percent of the Occupational Safety and Health Administration (OSHA) permissible exposure limit (0.1 f/cc). That is, action will be taken for any PCM result for TWA personal air samples of > 0.05 f/cc. Excursion samples are considered acceptable by PCM if the result is ≤ 50 percent of the OSHA permissible exposure limit (1.0 f/cc). That is, action will be taken for any PCM result for excursion samples of > 0.5 f/cc.

Corrective action: If either of the evaluation criteria is not met, then the CSF will be re-cleaned by wet-wiping and vacuuming the affected area and then ambient air samples will be recollected.

Results: No personal air PCM results were above the evaluation criteria. Therefore, no corrective action was taken based on these results.

4.1.2 TEM

All three types of samples (i.e., ambient air, personal air, and microvacuum dust) were analyzed by TEM. The evaluation criteria, corrective action, and results for each

are presented below. All evaluation criteria and corrective actions outlined in this section are described in the Close Support Facility Soil Preparation Plan (CDM 2003).

Ambient

Evaluation criteria: Ambient air samples are considered acceptable if one or fewer Libby amphibole (LA) structures are detected

Corrective action: If the evaluation criterion is not met, then the CSF will be re-cleaned by wet-wiping and vacuuming the affected area and then ambient air samples will be recollected.

Results: No ambient air TEM results were above the evaluation criteria. Therefore, no corrective action was taken based on these results

Personal Sample Results

Evaluation criteria: The action level for TEM analysis is 0.10 LA structures per cubic centimeter (s/cc) for structures between 0.5 microns and 5 microns and 0.01 s/cc for structures > 5 microns.

Corrective action: If either of these criteria is not met, the laboratory will be wet-wiped and HEPA vacuumed and ambient air samples recollected.

Results: No personal air TEM results exceeded the action level for structures between 0.5 microns and 5 microns. Two personal air samples (CS-11341 [sample coord.] and CS-11343 [sample prep.]) had results above the action level for structures > 5 microns. The LA concentration for CS-11341 and CS-11343 were 0.013 s/cc and 0.013 s/cc, respectively.

Microvacuum Results

Evaluation criteria: Microvacuum dust samples are considered acceptable if the TEM result is ≤ 5000 LA structures/cm^2.

Corrective action: If dust sample results indicate concentrations greater than 5,000 S/cm^2, the area represented by the sample will be wet-wiped, HEPA vacuumed, and re-sampled.

Results: The four microvacuum dust samples contained LA. However, only one sample (CS-11363 [office]) had a concentration greater than the action level (Figure 2-1). The concentration for CS-11363 was 7,900 S/cm^2.

4.2 Corrective Actions

A meeting was held on June 12, 2003 following the receipt of the TEM results, with CSF staff, health and safety (H&S) staff, field staff, and management to discuss the results of the negative exposure assessment and review the necessary corrective actions. Based on the analytical sample results and corrective action plans the laboratory needed to be wet-wiped, HEPA vacuumed, and samples recollected. However, CDM determined at this stage more rigorous cleaning was warranted.

Therefore, it was decided that thorough cleaning of the entire laboratory followed by aggressive air sampling would be performed.

4.2.1 June 18th Aggressive Air Sampling

Soil sample processing operations were placed on hold June 13. The CSF was subsequently cleaned June 16 through June 17. Aggressive air sampling was then conducted June 18 in accordance with EPA standard operating procedure (SOP) #2015 (EPA 1994). Based on the volume of the CSF, five box fans were used to move air for the sampling event (Figure 4-1). Five ambient air samples were collected using high-volume pumps at flow rates ranging between 8.42 and 8.58 L/min and analyzed for by TEM. The action level was considered zero LA fibers for all samples.

Table 4-1. June 18th Aggressive Air Sampling Sample Collection Key

Index Id	Sample Type	Personnel/Area	Duration (min)	Volume (L)
CS-11421	Ambient air	Receiving	180	1537.2
CS-11422	Ambient air	Main lab	180	1519.2
CS-11423	Ambient air	Receiving	180	1533.6
CS-11424	Ambient air	Soil drying	180	1537.2
CS-11425	Ambient air	Main lab	180	1537.2
CS-11426	Ambient air	Blank	180	n/a
CS-11427	Ambient air	Blank	180	n/a

L – liter min = minute n/a – not applicable

The results of the June 18th clearance sampling are presented in Table 4-2.

Table 4-2 Summary of June 18th Aggressive Air Sampling TEM Results

ID Number	Sample Type	Sampling Date	Personnel/Area	Asbestos Concentration* (s/cc)	Asbestos Mineral
CS-11421	Ambient air	6/18/03	Receiving	0.0388	Chrysotile
CS-11422	Ambient air	6/18/03	Main lab	<0.0023	ND
CS-11423	Ambient air	6/18/03	Receiving	0.0023	Chrysotile
CS-11424	Ambient air	6/18/03	Soil drying	0.0023	Chrysotile
CS-11425	Ambient air	6/18/03	Main lab	0.0045	Chrysotile/LA
CS-11426	Ambient air	6/18/03	Blank	ND	ND
CS-11427	Ambient air	6/18/03	Blank	ND	ND

ND = none detected
Filter Material = Mixed Cellulose Ester
LA = Libby amphibole
Average Grid Opening = 0.0110 mm^2
Filter Diameter = 25 mm
s/cc = structures per cubic centimeter
NA = not applicable
Effective Filter Area = 385 mm^2
* Total asbestos concentration

4.2.2 June 20th Aggressive Air Sampling

The June 18th aggressive air-sampling event found one LA structure on sample number CS-11425 (Main laboratory) (Figure 4-1). As a result, the CSF was re-cleaned and re-sampled in accordance with EPA SOP #2015 (EPA 1994). Once again, five ambient air monitors along with five box fans were used for the sampling event (Figure 4-1). Samples were collected using high-volume pumps at flow rates ranging between 8.42 and 8.58 L/min and analyzed by TEM.

Table 4-3. June 20th Aggressive Air Sampling Sample Collection Key

Index ID	Sample Type	Personnel/Area	Duration (min)	Volume (L)
CS-11428	Ambient air	Receiving	180	1533.6
CS-11429	Ambient air	Receiving	180	1539
CS-11430	Ambient air	Soil drying	180	1533.6
CS-11405	Ambient air	Main lab	180	1544.4
CS-11406	Ambient air	Main lab	180	1539
CS-11408	Ambient air	Blank	180	n/a
CS-11409	Ambient air	Blank	180	n/a

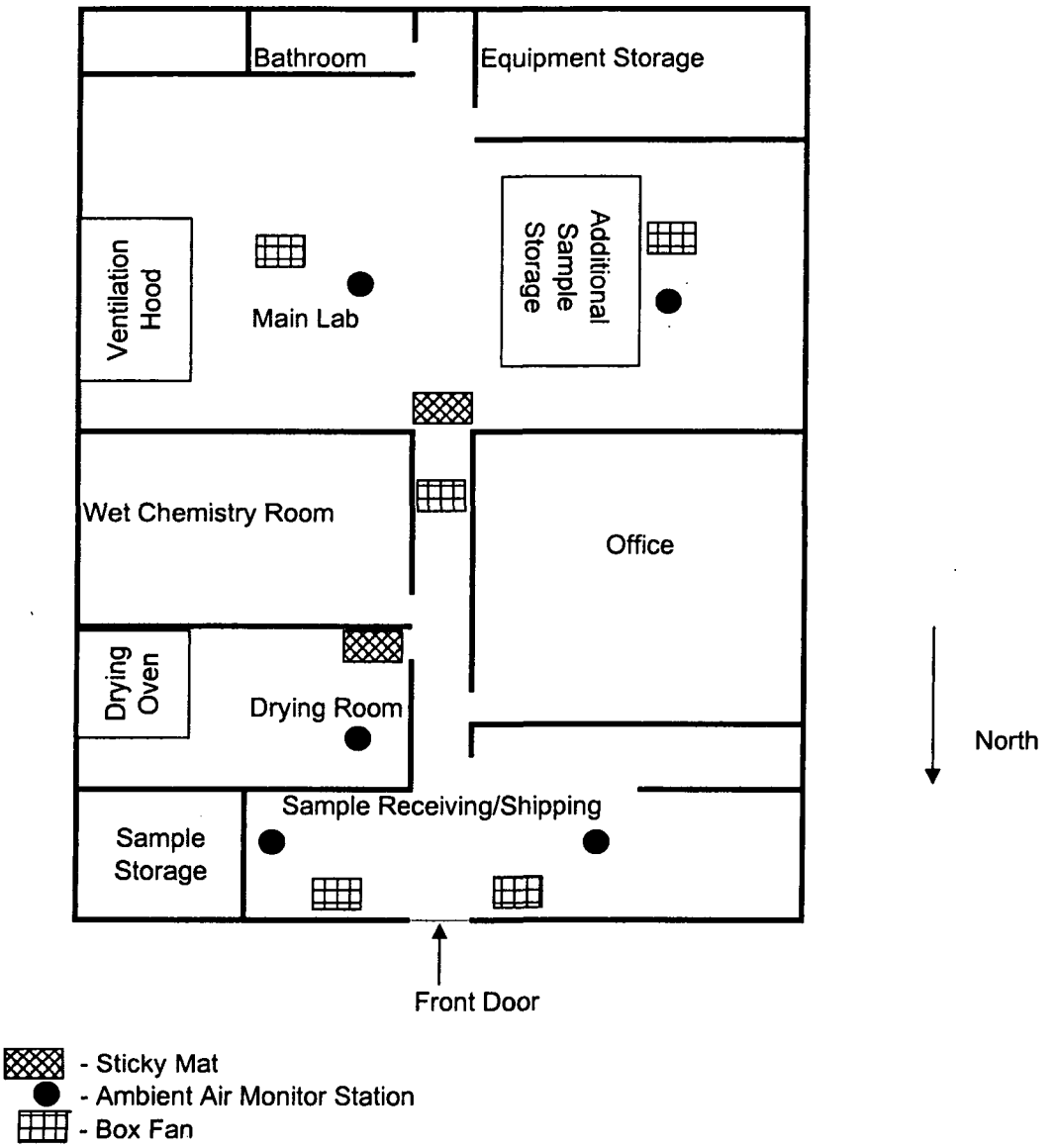
L = liters min = minutes

The results of the June 20th clearance sampling are presented in Table 4-4.

Table 4-4 Summary of June 20th Aggressive Air Sampling TEM Results

Index ID	Sample Type	Date	Personnel/Area	Asbestos Concentration (s/cc)
CS-11428	Ambient air	6/20/03	Receiving	<0.0046
CS-11429	Ambient air	6/20/03	Receiving	<0.0045
CS-11430	Ambient air	6/20/03	Soil drying	<0.0046
CS-11405	Ambient air	6/20/03	Main lab	<0.0045
CS-11406	Ambient air	6/20/03	Main lab	<0.0045
CS-11408	Ambient air	6/20/03	Blank	-
CS-11409	Ambient air	6/20/03	Blank	-

Figure 4-1 Aggressive Air Sampling Locations



Section 5
Conclusions

Findings from the negative exposure assessment indicated that there was the presence of a few LA structures in the CSF. Once those results were known, soil sample processing operations ceased. Aggressive air sampling was conducted on two occasions, June 18, 2003 and June 20, 2003. One LA fiber was detected during the first round of sampling and zero LA fibers were detected during the second round of sampling. At the time of this document, discussions are being held between EPA, Volpe, and CDM to determine if and what procedural changes need to be made to help ensure that future LA presence is prevented at the CSF.

Section 6
References

AHERA 2002. Asbestos Hazard Emergency Response Act 40 CFR (Protection of Environment) Chapter I (Environmental Protection Agency) Subchapter R (Toxic Substances Control Act) Part 763 (Asbestos) Subpart E (Asbestos Containing Materials in Schools) Appendix A (Interim Transmission Electron Microscopy Analytical Methods - Mandatory and Nonmandatory - and Mandatory Section to Determine Completion of Response Actions). Source is the Federal Register 2 FR 41846, October, 1987 Data current as of the Federal Register Dated May 20, 2002.

ASTM 1995. Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations. ASTM D-5755-95

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TARGET SHEET

EPA REGION VIII

SUPERFUND DOCUMENT MANAGEMENT SYSTEM

DOCUMENT NUMBER: 2009718

SITE NAME: LIBBY ASBESTOS

DOCUMENT DATE: 07/30/2003

DOCUMENT NOT SCANNED

Due to one of the following reasons:

- ☐ PHOTOGRAPHS
- ☐ 3-DIMENSIONAL
- ☐ OVERSIZED
- ☐ AUDIO/VISUAL
- ☐ PERMANENTLY BOUND DOCUMENTS
- ☐ POOR LEGIBILITY
- ☐ OTHER
- ☐ NOT AVAILABLE
- ☑ TYPES OF DOCUMENTS NOT TO BE SCANNED
  (Data Packages, Data Validation, Sampling Data, CBI, Chain of Custody)

DOCUMENT DESCRIPTION:

APPENDIX A Sample Bench Sheets

Contact the Superfund Records Center to view available document.
(303) 312-6473

Appendix B
U.S. EPA Standard Operating Procedure 2015. Asbestos Sampling

ASBESTOS SAMPLING

SOP#: 2015
DATE: 11/17/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

Asbestos has been use d in many commercial products including building materials such as flooring tiles and sheet goods, paints and coatings, insulation, an d roofing asphalts. These products and others may b e found at hazardous waste sites hanging on overhea d pipes, contained in drums, abandoned in piles, or a s part of a structure. Asbe stos tailing piles from mining operat ions can also be a source of ambient asbesto s fibers. Asbestos is a known carcinogen and requires air sampling to assess airborne exposure to huma n health. This Standard Operating Procedure (SOP) provides procedures for asbestos air sampling b y drawing a known volume of air through a mixe d cellulose ester (MCE) filter. The filter is then sent to a laboratory for analysis. The U.S. Environmenta l Protection Agency/Environmental Response Tea m (U.S. EPA/ERT) uses one of four analytical methods for determining asbestos in air. These include: U.S . EPA's Environmental Asbestos Assessment Manual, Superfund M ethod for the Determination of Asbestos in Ambient Air for Transmission Electron Microscopy (TEM)[1]; U.S. EPA's Modified Yamate Method fo r TEM[2]; National Institute f or Occupational Safety and Health (NIOSH) Method 7402 (direct method only) for TEM; and NIOSH Method 7400 for Phas e Contrast Microscopy (PCM) [3]. Each method ha s specific sampling and analytical requirements (i.e. , sample volume and flow rate) for determinin g asbestos in air.

The U.S. EPA/ERT typically follows procedure s outlined in the TEM methods for determinin g mineralogical types of asbestos in air and fo r distinguishing asbestos from non-asbestos minerals . The Phase Contrast Microscopy (PCM) method i s used by U.S. EPA/ERT as a screening tool since it is less costly than TEM. PCM cannot distinguis h asbestos from non-asbestos fibers, therefore the TEM metho d may be necessary to confirm analytica l results. For example, if an action level for th e presence of fibers has been set and PCM analysi s indicates that the act ion level has been exceeded, then TEM analysis can be used to quantify and identif y asbestos structures through examination of thei r morphology crystal structures (through electro n diffraction), and elemental composition (throug h energy dispersive X-ray analysis). In this instanc e samples should be collected for both analyses in side by side sam pling trains (some laboratories are able to perform PCM and TEM analysis from the same filter). The Superfund method is designed specifically t o provide results suitable for supporting ris k assess sments at Superfund sites, it is applicable to a wide range of ambient air situations at hazardou s waste sites. U.S. EPA's Modi fied Yamate Method for TEM is also used for ambient air sampling due to high volume requirements. The PCM and TEM NIOS H analytical methods require lower sample volumes and are typically used indoors; however, ERT wil l increase the volume requirement for outdoo r application.

Other Regulations pertaining to asbestos have bee n promulgated by U.S. EPA and OSHA. U.S. EPA' s National Emission Standards for Hazardous Ai r Pollutants (NESHAP) regulates asbestos-containin g waste materials. NESHAP establishes managemen t practices and standards for the handling of asbesto s and emissions from waste disposal operations (4 0 CFR Part 61, Subparts A and M). U.S. EPA's 40 CFR 763 (July 1, 1987) [4] and its addendum 40 CFR 76 3 (October 30, 1 987)[4] provide comprehensive rules for the asbestos abatement industry. State and loca l regula tions on these issues vary and may be mor e stringent than federal requirements. The OSH A regulations in 29 CFR 1910.1001 and 29 CF R 1926.58 specify w ork practices and safety equipment such as respiratory protection and protective clothing when handling asbestos. The OSHA standard for an 8-hour, time-weighted average (TWA) is 0. 2 fibers/cubic centimet ers of air. This standard pertains to fibers with a length-to-width ratio of 3 to 1 with a fiber length >5 μm [5,6]. An action level of 0.1 fiber/cc (one-half the OSHA standard) is the level U.S. EP A has established in which employers must initiate such activities as air monitoring, employee training, an d

medical surveillance [5,6].

These are standard (i.e., typically applicable) operating procedures w hich may be varied or changed as required, dependent upon site conditions , equipment limitations or limitations imposed by th e procedure. In all instances, the ultimate procedure s employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement o r recommendation for use.

2.0 METHOD SUMMARY

Prior to sampling, the site should be characterized by identifying on-site as well as off-site sources o f airborne asbestos. The array of sampling location s and the schedule for sample collection, is critical t o the success of an investigation. Generally, sampling strategies to characterize a single point source ar e fairly straightforward, while multiple point source s and area sources increase the complexity of th e sampling strategy. It is not within the scope of thi s SOP to provide a generic asbestos air sampling plan. Experience, objectives, and site characteristics wil l dictate the sampling strategy.

During a site investigation, sampling stations should be arranged to distinguish spatial trends in airborn e asbestos concentrations. Sampling schedules should be fashioned to establish temporal trends. Th e sampling strategy typically requires that th e concentration of asbest os at the source (worst case) or area of concern (downwind), crosswind, as well a s background (upwind) contribut ions be quantified. See Table 1 (Appendix A) for U.S. EPA/ER T recommended sampling set up for ambient air. Indoor asbestos sampling req uires a different type of strategy which is identified in Table 2 (Appendix A). It i s important to establish background levels o f contaminants in order to develop a reference poin t from which to evaluate the source data. Field blanks and lot blanks can be utilized to determine othe r sources.

Much information can be deri ved from each analytical method previously mentioned. Each analytica l method has specific sampling requirements an d produce results which may or may not be applicabl e to a specific sampling effort. The site samplin g objectives should be carefully id entified so as to select the most appropriate ana lytical method. Additionally, some preparation (i.e., lot blanks results) prior to site sampling may be required, these requirements ar e specified in the analytical methods.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

3.1 Sample Preservation

No preservation is required for asbestos samples.

3.2 Sample Handling, Container an d Storage Procedures

1. Place a sample label on the cassett e indicating a unique sampling number. D o not put sampling cassettes in shirt or coa t pockets as the filter can pick up fibers. The original cassette box is used to hold th e samples.

2. Wrap the cassette individually in a plasti c sample bag. Each bag should be marke d indicating sample identifica tion number, total volume, and date.

3. The wrapped sampling cassettes should b e placed upright in a rigid container so that the cassette cap is on top and cassette base is on bottom. Use enough packing material t o prevent jostling or damage. Do not us e vermiculite as packing material for samples. If possible, hand carry to lab.

4. Provide appropriate documentation wit h samples (i.e., chain of custody and requested analytical methodology).

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Flow rates exceeding 16 liters/minute (L/min) which could result in filter destructi on due to (a) failure of its physical support under force from the increase d pressure drop; (b) leakage of air around the filte r mount so that the filter is bypassed, or (c) damage to the asbestos structures due to increased impac t velocities.

4.1 U.S. EPA's Superfund Method

4.1.1 Direct-transfer TEM Specimen Preparation Methods

Direct-Transfer TEM specimen preparation methods have the following significant interferences:

- The achievable detection limit is restricted by the particulate density on the filter, which in turn is controlled by the sampled air volume and the total suspended particulate concentration in the atmosphere being sampled.
- The precision of the result is dependent on the uniformity of the deposit of asbestos structures on the sample collection filter.
- Air samples must be collected so that they have particulate and fiber loadings within narrow ranges. If too high a particulate loading occurs on the filter, it is not possible to prepare satisfactory TEM specimens by a direct-transfer method. If too high a fiber loading occurs on the filter, even if satisfactory TEM specimens can be prepared, accurate fiber counting will not be possible.

4.1.2 Indirect TEM Specimen Preparation Methods

Indirect TEM specimen preparation methods have the following interferences:

- The size distribution of asbestos structures is modified.
- There is increased opportunity for fiber loss or introduction of extraneous contamination.
- When sample collection filters are ashed, any fiber contamination in the filter medium is concentrated on the TEM specimen grid.

It can be argued that direct methods yield an under-estimate of the asbestos structure concentration because many of the asbestos fibers present are concealed by other particulate material with which they are associated. Conversely, indirect methods can be considered to yield an over-estimate because some types of complex asbestos structures disintegrate during the preparation, resulting in an increase in the numbers of structures counted.

4.2 U.S. EPA's Modified Yamate Method for TEM

High concentrations of background dust interfere with fiber identification.

4.3 NIOSH Method for TEM

Other amphibole particles that have aspect ratios greater than 3:1 and elemental compositions similar to the asbestos minerals may interfere in the TEM analysis. Some non-amphibole minerals may give electron diffraction patterns similar to amphiboles. High concentrations of background dust interfere with fiber identification.

4.4 NIOSH Method for PCM

PCM cannot distinguish asbestos from non-asbestos fibers; therefore, all particles meeting the counting *criteria are counted as total asbestos fibers. Fiber less* than 0.25 um in length will not be detected by this method. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

5.0 EQUIPMENT/MATERIALS

5.1 Sampling Pump

The constant flow or critical orifice controlled sampling pump should be capable of a flow-rate and pumping time sufficient to achieve the desired volume of air sampled.

The lower flow personal sampling pumps generally provide a flow rate of 20 cubic centimeters/minute (cc/min) to 4 L/min. These pumps are usually battery powered. High flow pumps are utilized when flow rates between 2 L/min to 20 L/min are required. High flow pumps are used for short sampling periods so as to obtain the desired sample volume. High flow pumps usually run on AC power and can be plugged into a nearby outlet. If an outlet is not available then a generator should be obtained. The generator should be positioned downwind from the sampling pump. Additional voltage may be required if more than one pump is plugged into the same generator. Several

electrical extension cord s may be required if sampling locations are remote.

The recommended volume for the Superfund method (Phase I) requires approximately 20 hours to collect. Such pumps typically draw 6 amps at full power s o that 2 lead/acid batteries should provide sufficien t power to collect a full sample. The use of lin e voltage, where available, eliminates the difficultie s associated with transporting stored electrical energy.

A stand should be used to hold th e filter cassette at the desired height for sampling and t he filter cassette shall be isolated from the vibrations of the pump.

5.2 Filter Cassette

The cassettes are purchased wit h the required filters in position, or can be assembled in a laminar flow hood or clean area. When the filters are in position, a shrink cellulose band or adhesive tape should b e applied to cassette joints to prevent air leakage.

5.2.1 TEM Cassette Requirements

Commercially available field monitors, comprisin g 25 mm diameter three-piece cassettes, wit h conductive extension cowls shall be used for sample collection. The cassette must be new and no t previously used. The cassette shall be loaded with an MCE filter of pore size 0.45 µm, and supplied from a lot number which has been qualified as lo w background for asbestos determination. The cowl s should be constructed of electrically conductin g material to minimize electrostatic effects. The filte r shall be backed by a 5 µm pore size MCE filte r (Figure 1, Appendix B).

5.2.2 PCM Cassette Requirements

NIOSH Method 7400, PCM involves using a 0.8 t o 1.2 µm mixed cellulose ester membrane, 25 m m diameter, 50 mm conductive cowl on cassette (Figure 2, Appendix B). Some labs are able to perform PCM and TEM analysis on the same filter; however, thi s should be discussed with the laboratory prior t o sampling.

5.3 Other Equipment

- Inert tubing with glass cyclone and hose barb
- Whirlbags (plastic bags) for cassettes
- Tools - small screw drivers
- Container - to keep samples upright
- Generator or electrical outlet (may not b e required)
- Extension cords (may not be required)
- Multiple plug outlet
- Sample labels
- Air data sheets
- Chain of Custody records

6.0 REAGENTS

Reagents are not required for the preservation o f asbestos samples.

7.0 PROCEDURES

7.1 Air Volumes and Flow Rates

Sampling volumes are deter mined on the basis of how many fibers need to be collected for reliabl e measurements. Therefore, one must estimate ho w many airborne fibers m ay be in the sampling location.

Since the concentration of airborne aeroso l contaminants will have some effect on the sample, the following is a suggested criteria to assist in selecting a flow rate based on rea l-time aerosol monitor (RAM) readings in milligrams/cubic meter (mg/m ^{3}).

	Concentration	Flow Rate
• Low RAM readings:	<6.0 mg/m ^{3}	11-15. L/min
• Medium RAM readings:	>6.0 mg/m ^{3}	7.5 L/min
• High RAM readings:	>10. mg/m ^{3}	2.5 L/min

In practice, pumps that are availab le for environmental sampling at remote locations operate under a maximum load of approximately 12 L/min.

7.1.1 U.S. EPA's Superfund Method

The Superfund Method incorporates an indirec t preparation procedure to provide flexibility in th e amount of deposit that be can be tolerated on th e sample filter and to allow for the selectiv e concentration of asbestos prior to analysis. T o minimize contributions to background contamination from asbestos present in the plastic matrices o f membrane filters while allowing for sufficien t quantities of asbestos to be collected, this method also requires the collection of a larger volume of air pe r unit area of fi lter than has traditionally been collected

for asbestos analysis. Due to the need to collect large volumes of air, higher sampling flow rates are recommended in this method than have generally been employed for asbestos sampling in the past. As an alternative, samples may be collected over longer time intervals. However, this restricts the flexibility required to allow samples to be collected while uniform meteorological conditions prevail.

The sampling rate and the period of sampling should be selected to yield as high a sampled volume as possible, which will minimize the influence of filter contamination. Wherever possible, a volume of 15 cubic meters (15,000 L) shall be sampled for those samples intended for analysis only by the indirect TEM preparation method (Phase 1 samples). For those samples to be prepared by both the indirect and the direct specimen preparation methods (Phase 2 samples), the volumes must be adjusted so as to provide a suitably-loaded filter for the direct TEM preparation method. One option is to collect filters at several loadings to bracket the estimated optimum loading for a particular site. Such filters can be screened in the laboratory so that only those filters closest to optimal loading are analyzed. It has been found that the volume cannot normally exceed 5 cubic meters (5000 L) in an urban or agricultural area, and 10 cubic meters (10,000 L) in a rural area for samples collected on a 25 mm filter and prepared by a direct-transfer technique.

An upper limit to the range of acceptable flow rates for this method is 15 L/min. At many locations, wind patterns exhibit strong diurnal variations. Therefore, intermittent sampling (sampling over a fixed time interval repeated over several days) may be necessary to accumulate 20 hours of sampling time over constant wind conditions. Other sampling objectives also may necessitate intermittent sampling. The objective is to design a sampling schedule so that samples are collected under uniform conditions throughout the sampling interval. This method provides for such options. Air volumes collected on Phase I samples are maximized (<16 L/min). Air volumes collected on Phase 2 samples are limited to provide optimum loading for filters to be prepared by a direct-transfer procedure.

7.1.2 U.S. EPA's Modified Yamate Method for TEM

U.S. EPA's TEM method requires a minimum volume of 560 L and a maximum volume of 3,800 L in order to obtain an analytical sensitivity of 0.005 structures/cc. The optimal volume for TEM is 1200 L to 1800 L. These volumes are determined using a 200 mesh EM grid opening with a 25-mm filter cassette. Changes in volume would be necessary if a 37-mm filter cassette is used since the effective area of a 25 mm (385 sq mm) and 37 mm (855 sq m) differ.

7.1.3 NIOSH Method for TEM and PCM

The minimum recommended volume for TEM and PCM is 400 L at 0.1 fiber/cc. Sampling time is adjusted to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for eight hours (700 to 2800 L) is appropriate in non-dusty atmospheres containing 0.1 fiber/cc. Dusty atmospheres i.e., areas with high levels of asbestos, require smaller sample volumes (<400 L) to obtain countable samples.

In such cases, take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3,000 to 10,000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If > 50% of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration. Do not exceed 0.5 mg total dust loading on the filter.

7.2 Calibration Procedures

In order to determine if a sampling pump is measuring the flow rate or volume of air correctly, it is necessary to calibrate the instrument. Sampling pumps should be calibrated immediately before and after each use. Preliminary calibration should be conducted using a primary calibrator such as a soap bubble type calibrator, (e.g., a Buck Calibrator, Gilibrator, or equivalent primary calibrator) with a representative filter cassette installed between the pump and the calibrator. The representative sampling cassette can be reused for calibrating other pumps that will be used for asbestos sampling. The same cassette lot used for sampling should also be used for the calibration. A sticker should be affixed to the outside of the extension cowl marked "Calibration Cassette."

A rotameter can be used provided it has been recently precalibrated with a primary calibrator. Three separate constant flow calibration readings should be obtained both before sampling and after sampling. Should the flow rate change by more than 5% during the sampling period, the average of the pre- and post-calibration rates will be used to calculate the total sample volume. The sampling pump used shall provide a non-fluctuating air-flow through the filter, and shall maintain the initial volume flow-rate to within ± 10% throughout the sampling period. The mean value of these flow-rate measurements shall be used to calculate the total air volume sampled. A constant flow or critical orifice controlled pump meets these requirements. If at any time the measurement indicates that the flow-rate has decreased by more than 30%, the sampling shall be terminated. Flexible tubing is used to connect the filter cassette to the sampling pump.Sampling pumps can be calibrated prior to coming on-site so that time is saved when performing on-site calibration.

7.2.1 Calibrating a Personal Sampling Pump with an Electronic Calibrator

1. See Manufacturer's manual for operational instructions.

2. Set up the calibration train as shown in (Figure 3, Appendix B) using a sampling pump, electronic calibrator, and a representative filter cassette. The same lot sampling cassette used for sampling should also be used for calibrating.

3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 foot) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the electronic calibrator.

4. Turn the electronic calibrator and sampling pump on. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

5. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

6. Perform the calibration three times until the desired flow rate of ± 5% is attained.

7.2.2 Calibrating a Rotameter with an Electronic Calibrator

1. See manufacturer's manual for operational instructions.

2. Set up the calibration train as shown in (Figure 4, Appendix B) using a sampling pump, rotameter, and electronic calibrator.

3. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6° vertical.

4. Turn the electronic calibrator and sampling pump on.

5. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

6. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

7. Record the electronic calibrator flow rate reading and the corresponding rotameter reading. Indicate these values on the rotameter (sticker). The rotameter should be able to work within the desired flow range. Readings can also be calibrated for 10 cm^3 increments for Low Flow rotameters, 500 cm^3 increments for medium flow rotameters and 1 liter increments for high flow rotameters.

8. Perform the calibration three times until the desired flow rate of ± 5% is attained. Once on site, a secondary calibrator, i.e., rotameter may be used to calibrate sampling pumps.

7.2.3 Calibrating a Personal Sampling Pump with a Rotameter

1. See manufacturer's manual for Rotameter's Operational Instructions.

2. Set up the calibration train as shown in (Figure 5, Appendix B) using a rotameter, sampling pump, and a representative sampling cassette.

3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 ft) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the rotameter.

4. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6° vertical.

5. Turn the sampling pump on.

6. Turn the flow adjust screw (or knob) on the personal sampling pump until the float ball on the rotameter is lined up with the precalibrated flow rate value. A sticker on the rotameter should indicate this value.

7. A verification of calibration is generally performed on-site in the clean zone immediately prior to the sampling.

7.3. Meteorology

It is recommended that a meteorological station be established. If possible, sample after two to three days of dry weather and when the wind conditions are at 10 mph or greater. Record wind speed, wind direction, temperature, and pressure in a field logbook. Wind direction is particularly important when monitoring for asbestos downwind from a fixed source.

7.4 Ambient Sampling Procedures

7.4.1 Pre-site Sampling Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.

2. Obtain necessary sampling equipment and ensure it is in working order and fully charged (if necessary).

3. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety plan.

4. Once on-site the calibration is performed in the clean zone. The calibration procedures are listed in Section 7.2.

5. After calibrating the sampling pump, mobilize to the sampling location.

7.4.2 Site Sampling

1. To set up the sampling train, attach the air intake hose to the cassette base. Remove the cassette cap (Figure 6 and 7, Appendix B). The cassette should be positioned downward, perpendicular to the wind

2. If AC or DC electricity is required then turn it on. If used, the generator should be placed 10 ft. downwind from the sampling pump.

3. Record the following in a field logbook: date, time, location, sample identification number, pump number, flow rate, and cumulative time.

4. Turn the pump on. Should intermittent sampling be required, sampling filters must be covered between active periods of sampling. To cover the sample filter: turn the cassette to face upward, place the cassette cap on the cassette, remove the inlet plug from the cassette cap, attach a rotameter to the inlet opening of the cassette cap to measure the flow rate, turn off the sampling pump, place the inlet plug into the inlet opening on the cassette cap. To resume sampling: remove the inlet plug, turn on the sampling pump, attach a rotameter to measure the flow rate, remove the cassette cap, replace the inlet plug in the cassette cap and invert the cassette, face downward and perpendicular to the wind.

5. Check the pump at sampling midpoint if sampling is longer than 4 hours. The generators may need to be regased depending on tank size. If a filter darkens in appearance or if loose dust is seen in the filter, a second sample should be started.

6. At the end of the sampling period, orient the cassette up, turn the pump off.

7. Check the flow rate as shown in Section 7.2.3. When sampling open-faced, the sampling cap should be replaced before post calibrating. Use the same cassette used for sampling for post calibration (increase dust/fiber loading may have altered the flow rate.

8. Record the post flow rate.

9. Record the cumulative time or run.

10. Remove the tubing from the sampling cassette. Still holding the cassette upright, replace the inlet plug on the cassette cap and the outlet plug on the cassette base.

7.4.3. Post Site Sampling

1. Follow handling procedures in Section 3.2, steps 1-4.

2. Obtain an electronic or hard copy of meteorological data which occurred during the sampling event. Record weather: wind speed, ambient temperature, wind direction, and precipitation. Obtaining weather data several days prior to the sampling event can also be useful.

7.5 Indoor Sampling Procedures

PCM analysis is used for indoor air samples. When analysis shows total fiber count above the OSHA action level 0.1 f/cc then TEM (U.S. EPA's Modified Yamate Method) is used to identify asbestos from non-asbestos fibers.

Sampling pumps should be placed four to five feet above ground level away from obstructions that may influence air flow. The pump can be placed on a table or counter. Refer to Table 2 (Appendix A) for a summary of indoor sampling locations and rationale for selection.

Indoor sampling utilizes high flow rates to increased sample volumes (2000 L for PCM and 2800 to 4200 L for TEM) in order to obtain lower detection limits below the standard, (i.e., 0.01 f/cc or lower [PCM] and 0.005 structures/cc or lower [TEM]).

7.5.1 Aggressive Sampling Procedures

Sampling equipment at fixed locations may fail to detect the presence of asbestos fibers. Due to limited air movement, many fibers may settle out of the air onto the floor and other surfaces and may not be captured on the filter. In the past, an 8-hour sampling period was recommended to cover various air circulation conditions. A quicker and more effective way to capture asbestos fibers is to circulate the air artificially so that the fibers remain airborne during sampling. The results from this sampling option typifies worst case condition. This is referred to as aggressive air sampling for asbestos. Refer to Table 2 for sample station locations.

1. Before starting the sampling pumps, direct forced air (such as a 1-horsepower leaf blower or large fan) against walls, ceilings, floors, ledges, and other surfaces in the room to initially dislodge fibers from surfaces. This should take at least 5 minutes per 1000 sq. ft. of floor.

2. Place a 20-inch fan in the center of the room. (Use one fan per 10,000 cubic feet of room space.) Place the fan on slow speed and point it toward the ceiling.

3. Follow procedures in Section 7.4.1 and 7.4.2 (Turn off the pump and then the fan(s) when sampling is complete.).

4. Follow handling procedures in Section 3.2, steps 1-4.

8.0 CALCULATIONS

The sample volume is calculated from the average flow rate of the pump multiplied by the number of minutes the pump was running (volume = flow rate X time in minutes). The sample volume should be submitted to the laboratory and identified on the chain of custody for each sample (zero for lot, field and trip blanks).

The concentration result is calculated using the sample volume and the numbers of asbestos structures reported after the application of the cluster and matrix counting criteria.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

Follow all QA/QC req uirements from the laboratories as well as the analytical methods.

9.1 TEM Requirements

1. Examine lot blanks to determine th e background asbes tos structure concentration.

2. Examine field blanks to determine whethe r there is contamination by extraneou s asbestos structures during specime n preparation.

3. Examine of labor atory blanks to determine if contamination is being introduced durin g critical phases of the laboratory program.

4. To determine if the laboratory ca n satisfactorily analyze samples of know n asbestos structure concentrations, referenc e filters shall be examined. Reference filter s should be maintained as part of th e laboratory's Quality Assurance program.

5. To minimize subjective effects, som e specimens should be r ecounted by a different microscopist.

6. Asbestos laboratories shall be accredited by the National Voluntary Laborator y Accreditation Program.

7. At this time, performance evaluation samples for asbestos in air are not available fo r Removal Program Activities.

9.2 PCM Requirements

1. Examine reference slides of know n concentration to determine the analyst' s ability to satisfactorily count fibers . Reference slides should be maintained a s part of the laboratory's quality assuranc e program.

2. Examine fi eld blanks to determine if there is contamination by extraneous structure s during sample handling.

3. Some samples should be relabeled the n submitted for counting by th e same analyst to determine possible bias by the analyst.

4. Participation in a profic iency testing program such as the AIHA-NIOSH proficienc y analytical testing (PAT) program.

10.0 DATA VALIDATION

Results of quality control samples will be evaluate d for contamination. This information will be utilize d to qualify the environmental sample result s accordingly with the project's data quality objectives.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials , follow U.S. EPA, OSHA, and corporate health an d safety procedures. More specifically, when entering an unknown situation involving asbestos, a powere d air purifying respirator (PAPR) (full face-piece) i s necessary in co njunction with HEPA filter cartridges. See applicable regulations for action level, PEL, TLV, etc. If previous sampling indicates asbesto s concentrations are below personal health and safet y levels, then Level D personal protection is adequate.

12.0 REFERENCES

(1) Environmental Asbestos Assessmen t Manual, Superfund Method for th e Determination of Asbestos in Ambient Air , Part 1: Method, EPA/540/2-90/005a, Ma y 1990, and Part 2: Technical Backgroun d Document, EPA/540/2-90/005b, May 1990.

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(5) U.S. Environmental Protection Agency. Asbestos-Containing Materials in Schools; Final Rule and Notice. 52 FR 41826.

(6) Occupational Safety and Health Administration. Code of Federal Regulations 29 CFR 1910.1001. Washington, D.C. 1987.

APPENDIX A

Tables

TABLE 1.

SAMPLE STATIONS FOR OUTDOOR SAMPLING

Sample Station Location	Sample Numbers	Rationale
Upwind/Background [1]	Collect a minimum of two simultaneous upwind/background samples 30 ° apart from the prevailing windlines.	Establishes background fiber levels.
Downwind	Deploy a minimum of 3 sampling stations in a 180 degree arc downwind from the source.	Indicates if asbestos is leaving the site.
Site Representative and/or Worst Case	Obtain one site representative sample which shows average condition on-site or obtain worst case sample (optional).	Verify and continually confirm and document selection of proper levels of worker protection.

[1] More than one background station may be required if the asbestos originates from different sources.

APPENDIX A (Cont'd)

Tables

TABLE 2

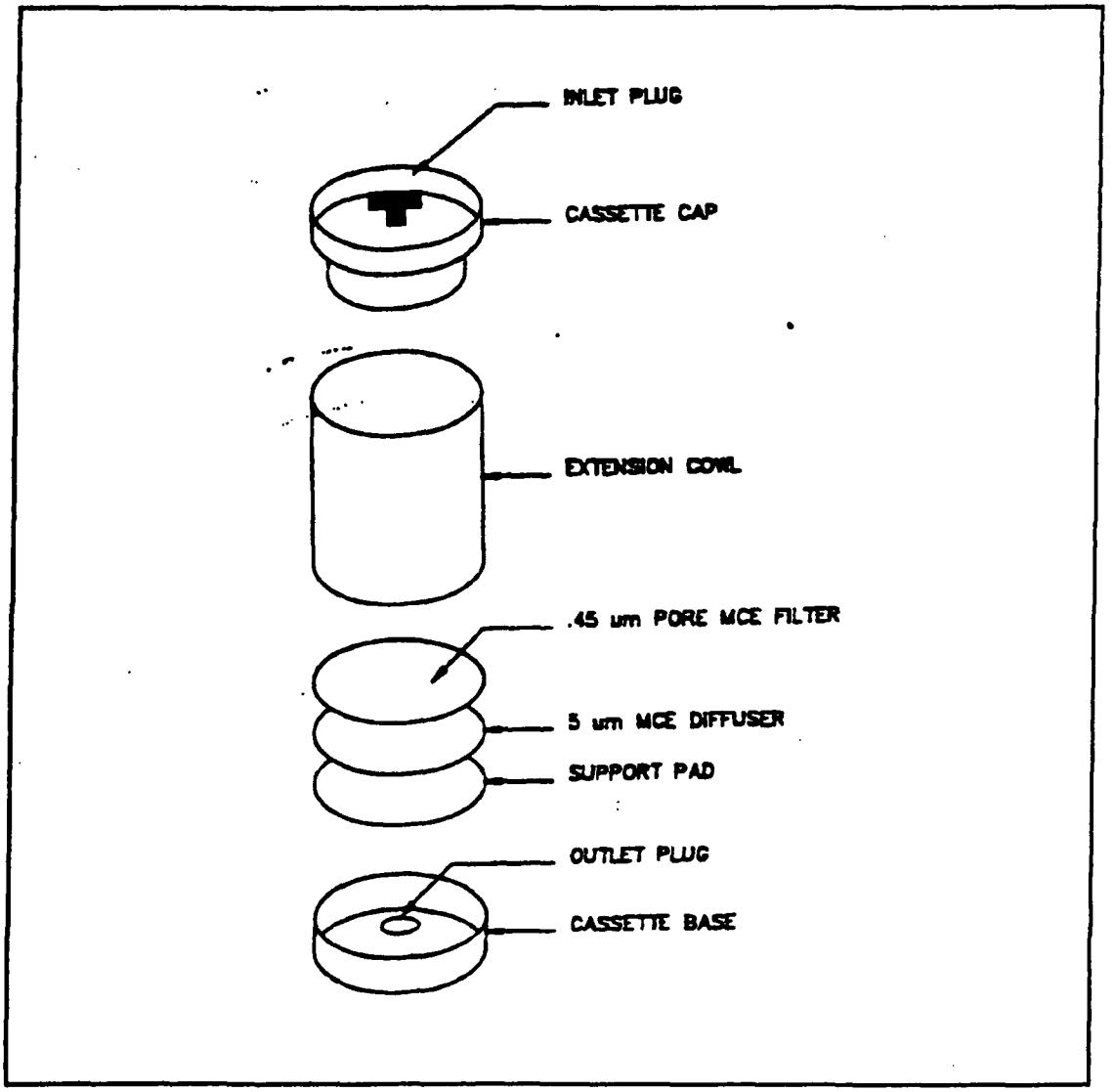
SAMPLE STATIONS FOR INDOOR SAMPLING

Sample Station Location	Sample Numbers	Rationale
Indoor Sampling	If a work site is a single room, disperse 5 samplers throughout the room. If the work site contains up to 5 rooms, place at least one sampler in each room. If the work site contains more than 5 rooms, select a representative sample of the rooms.	Establishes representative samples from a homogeneous area.
Upwind/Background	If outside sources are suspected, deploy a minimum of two simultaneous upwind/background samples 30 ° apart from the prevailing windlines.	Establish whether indoor asbestos concentrations are coming from an outside source.
Worst Case	Obtain one worst case sample, i.e., aggressive sampling (optional).	Verify and continually confirm and document selection of proper levels of worker protection.

APPENDIX B

Figures

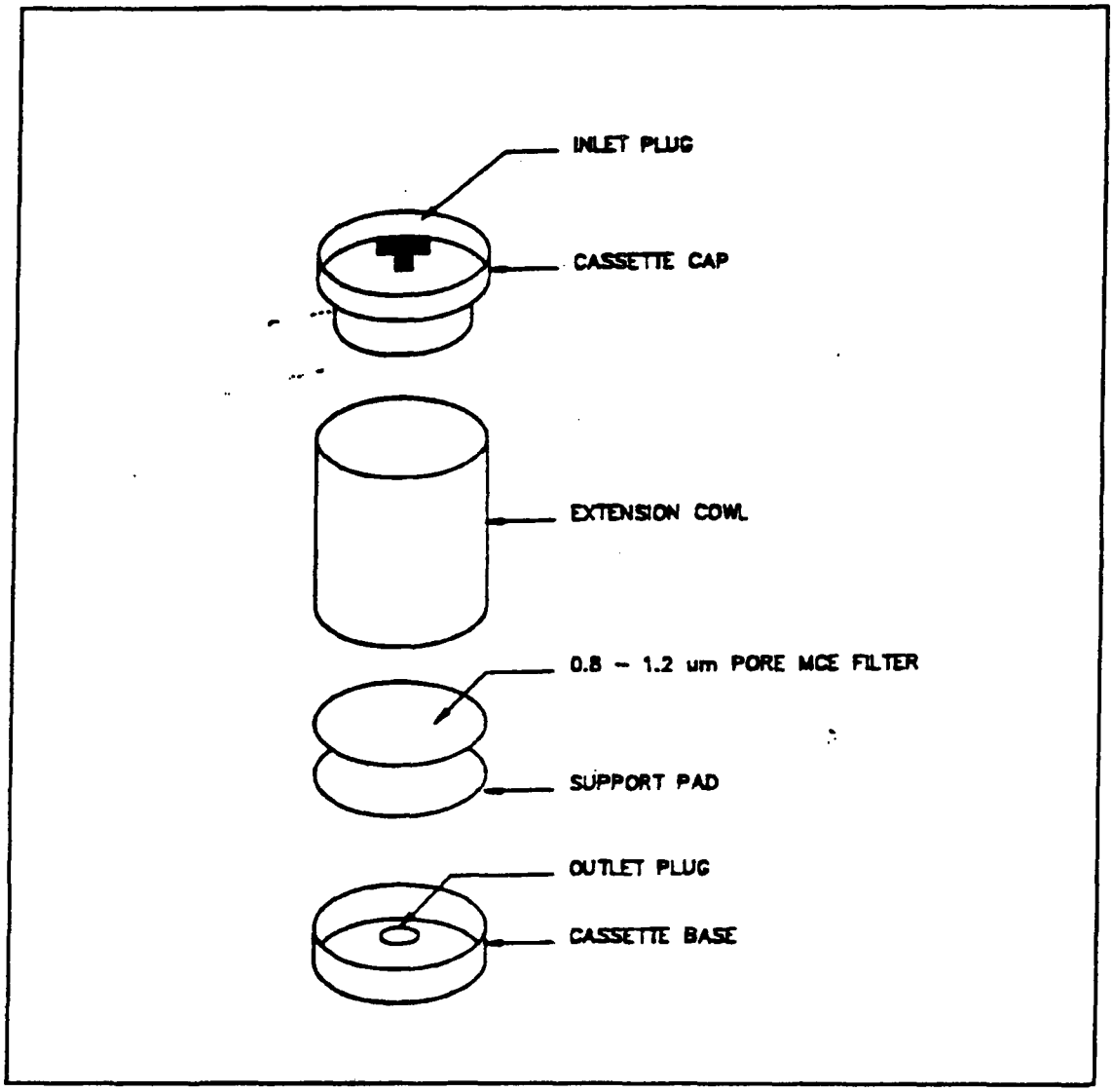
FIGURE 1. Transmission Electron Microscopy Filter Cassette



APPENDIX B (Cont'd)

Figures

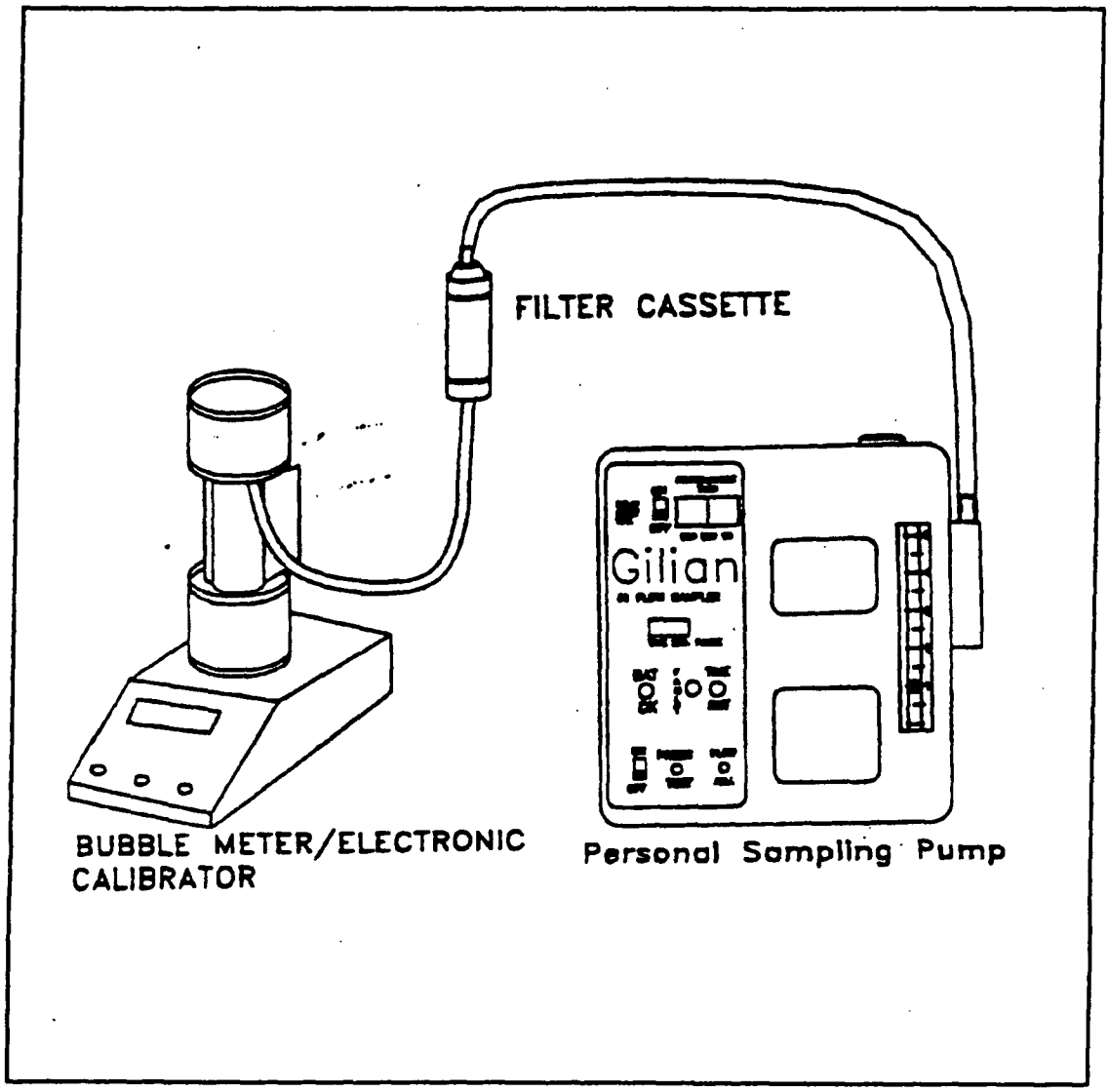
FIGURE 2. Phase Contrast Microscopy Filter Cassette



APPENDIX B (Cont'd)

Figures

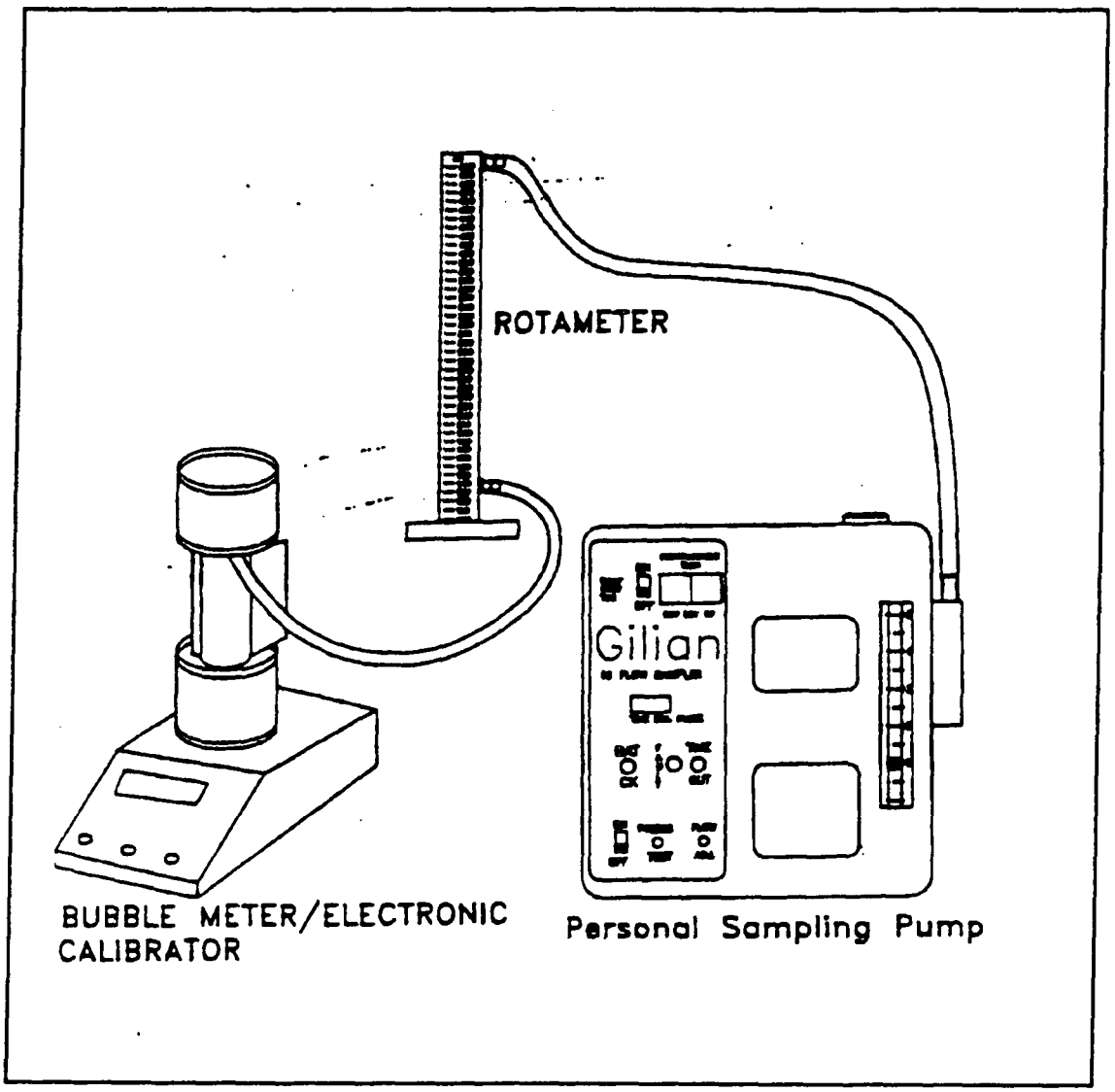
FIGURE 3. Calibrating a Personal Sampling Pump with a Bubble Meter



APPENDIX B (Cont'd)

Figures

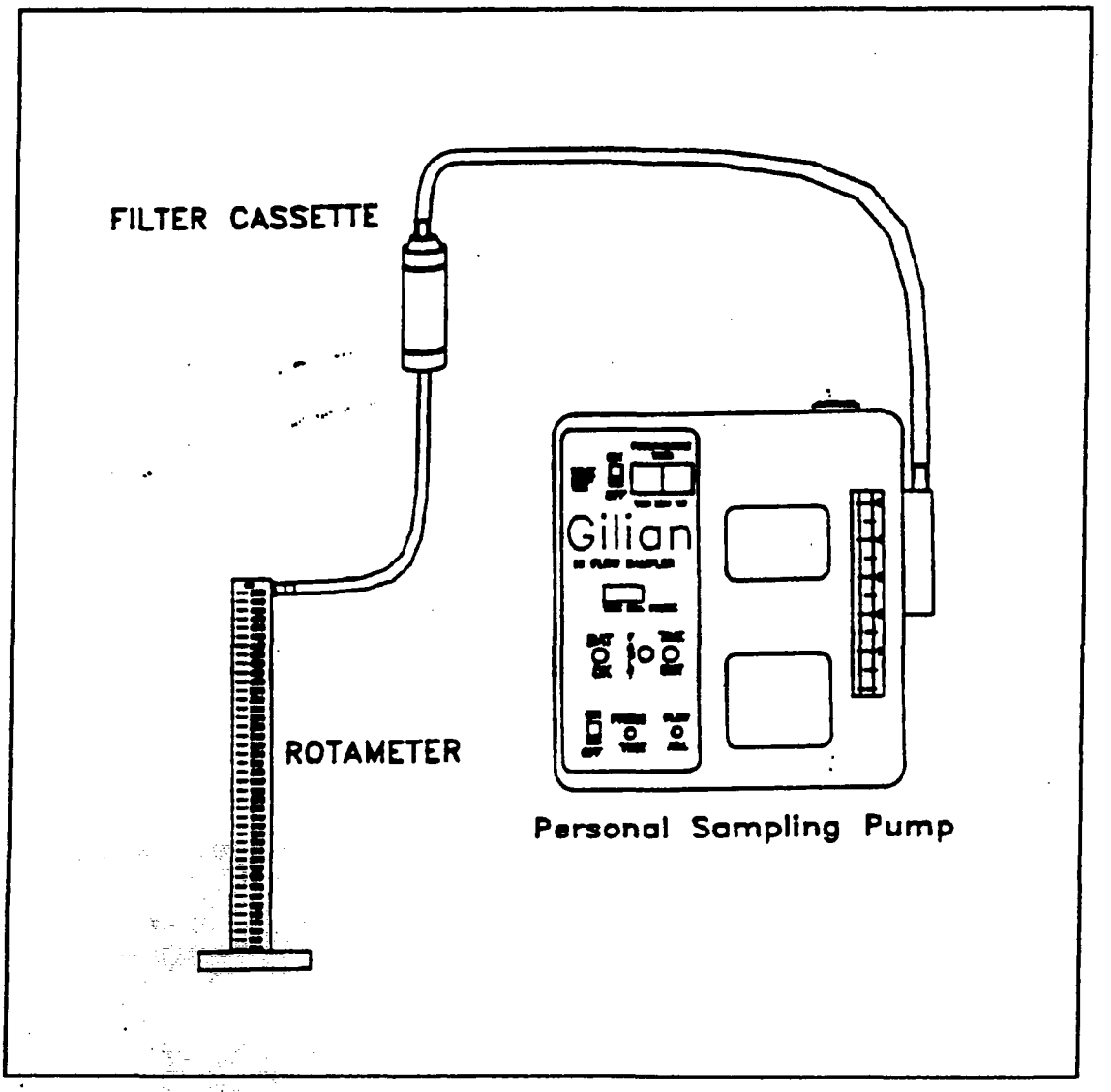
FIGURE 4. Calibrating a Rotameter with a Bubble Meter



APPENDIX B (Cont'd)

Figures

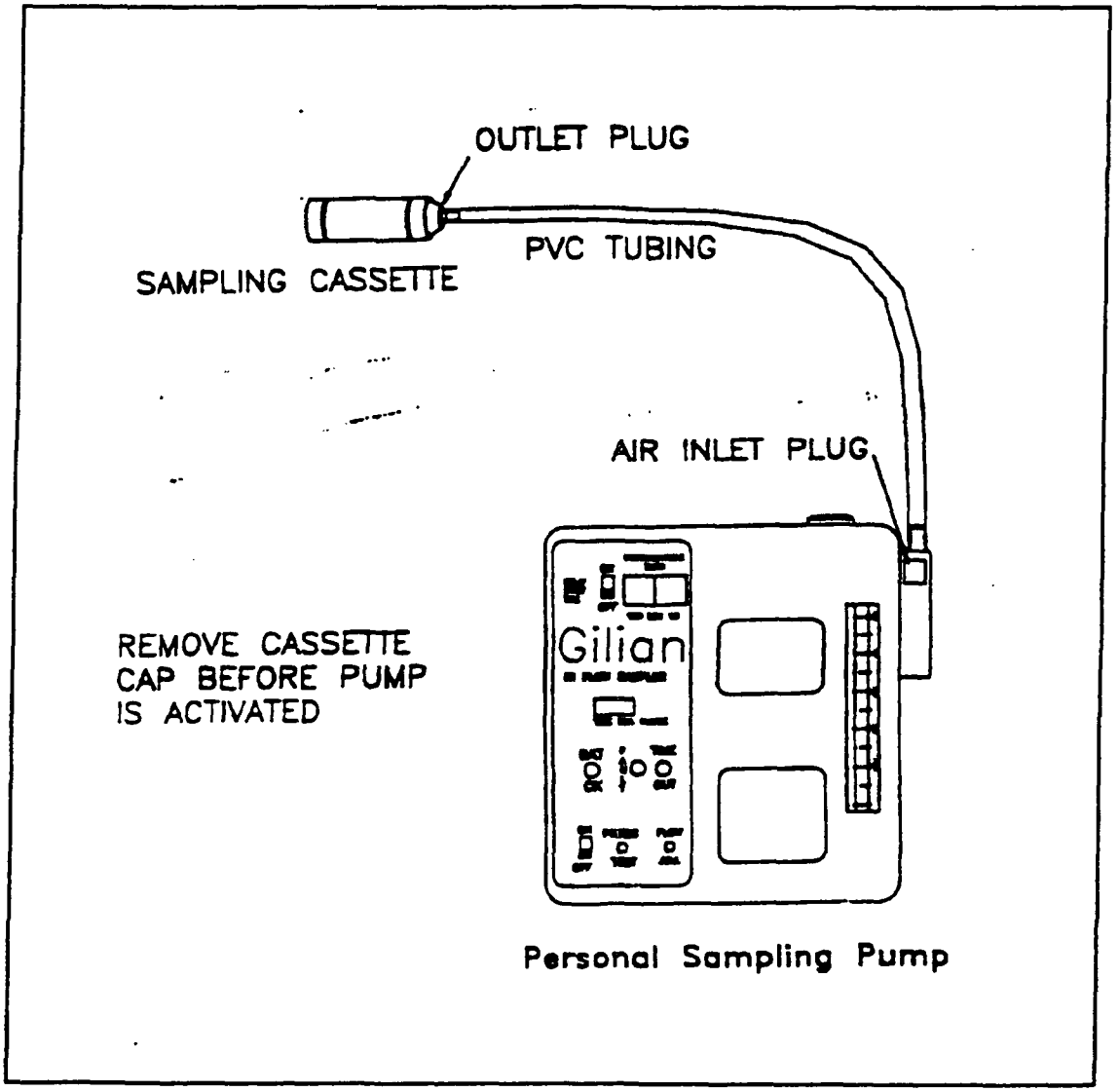
FIGURE 5. Calibrating a Sampling Pump with a Rotameter



APPENDIX B (Cont'd)

Figures

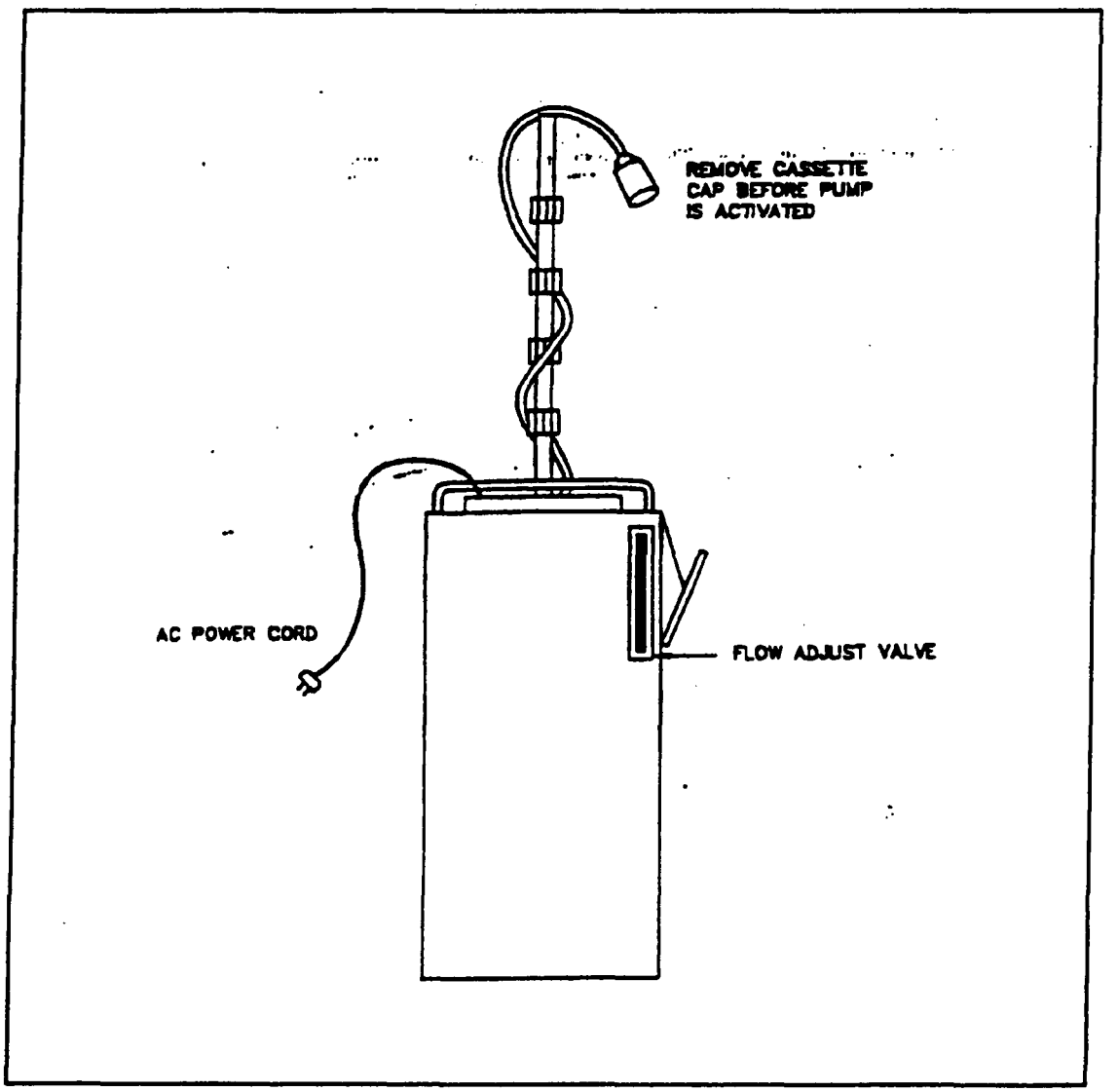
FIGURE 6. Personal Sampling Train for Asbestos



APPENDIX B (Cont'd)

Figures

FIGURE 7. High Flow Sampling Train for Asbestos



Appendix B
U.S. EPA Standard Operating Procedure 2015. Asbestos Sampling

ASBESTOS SAMPLING

SOP#: 2015
DATE: 11/17/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

Asbestos has been use d in many commercial products including building materials such as flooring tiles and sheet goods, paints and coatings, insulation, an d roofi ng asphalts. These products and others may b e found at hazardous waste sites hanging on overhea d pipes, contained in drums, abandoned in piles, or a s part of a structure. Asbe stos tailing piles from mining operat ions can also be a source of ambient asbesto s fibers. Asbestos is a known carcinogen and requires air sampling to assess airborne exposure to huma n health. This Standard Operating Procedure (SOP) provides procedures for asbestos air sampling b y drawing a known volume of air through a mixe d cellulose ester (MCE) filter. The filter is then sent to a laboratory for analysis. The U.S. Environmenta l Protection Agency/Environmental Response Tea m (U.S. EPA/ERT) uses one of four analytical methods for determining asbestos in air. These include: U.S . EPA's Environmental Asbestos Assessment Manual, Superfund M ethod for the Determination of Asbestos in Ambient Air for Transmission Electron Microscopy (TEM)[1]; U.S. EPA's Modified Yamate Method fo r TEM[2]; National Institute f or Occupational Safety and Health (NIOSH) Method 7402 (direct method only) for TEM; and NIOSH Method 7400 for Phas e Contrast Microscopy (PCM) [3]. Each method ha s specific sampling and analytical requirements (i.e. , sample volume and flow rate) for determinin g asbestos in air.

The U.S. EPA/ERT typically follows procedure s outlined in the TEM methods for determinin g mineralogical types of asbestos in air and fo r distinguishing asbestos from non-asbestos minerals . The Phase Contrast Microscopy (PCM) method i s used by U.S. EPA/ERT as a screening tool since it is less costly than TEM. PCM cannot distinguis h asbestos from non-asbestos fibers, therefore the TEM metho d may be necessary to confirm analytica l results. For example, if an action level for th e presence of fibers has been set and PCM analysi s indicates that the act ion level has been exceeded, then TEM analysis can be used to quantify and identif y asbestos structures through examination of thei r morphology crystal structures (through electro n diffraction), and elemental composition (throug h energy dispersive X-ray analysis). In this instanc e samples should be collected for both analyses in side by side sam pling trains (some laboratories are able to perform PCM and TEM analysis from the same filter). The Superfund method is designed specifically t o provide results suitable for supporting ris k assess sments at Superfund sites, it is applicable to a wide range of ambient air situations at hazardou s waste sites. U.S. EPA's Modi fied Yamate Method for TEM is also used for ambient air sampling due to high volume requirements. The PCM and TEM NIOS H analytical methods require lower sample volumes and are typically used indoors; however, ERT wil l increase the volume requirement for outdoo r application.

Other Regulations pertaining to asbestos have bee n promulgated by U.S. EPA and OSHA. U.S. EPA' s National Emission Standards for Hazardous Ai r Pollutants (NESHAP) regulates asbestos-containin g waste materials. NESHAP establishes managemen t practices and standards for the handling of asbesto s and emissions from waste disposal operations (4 0 CFR Part 61, Subparts A and M). U.S. EPA's 40 CFR 763 (July 1, 1987) [4] and its addendum 40 CFR 76 3 (October 30, 1 987)[4] provide comprehensive rules for the asbestos abatement industry. State and loca l regulations on these issues vary and may be mor e stringent than federal requirements. The OSH A regulations in 29 CFR 1910.1001 and 29 CF R 1926.58 specify w ork practices and safety equipment such as respiratory protection and protective clothing when handling asbestos. The OSHA standard for an 8-hour, time-weighted average (TWA) is 0. 2 fibers/cubic centimet ers of air. This standard pertains to fibers with a length-to-width ratio of 3 to 1 with a fiber length >5 μm [5,6]. An action level of 0.1 fiber/cc (one-half the OSHA standard) is the level U.S. EP A has established in which employers must initiate such activities as air monitoring, employee training, an d

medical surveillance [5,6].

These are standard (i.e., typically applicable) operating procedures w hich may be varied or changed as required, dependent upon site conditions , equipment limitations or limitations imposed by th e procedure. In all instances, the ultimate procedure s employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement o r recommendation for use.

2.0 METHOD SUMMARY

Prior to sampling, the site should be characterized by identifying on-site as well as off-site sources o f airborne asbestos. The array of sampling location s and the schedule for sample collection, is critical t o the success of an investigation. Generally, sampling strategies to characterize a single point source ar e fairly straightforward, while multiple point source s and area sources increase the complexity of th e sampling strategy. It is not within the scope of thi s SOP to provide a generic asbestos air sampling plan. Experience, objectives, and site characteristics wil l dictate the sampling strategy.

During a site investigation, sampling stations should be arranged to distinguish spatial trends in airborn e asbestos concentrations. Sampling schedules should be fashioned to establish temporal trends. Th e sampling strategy typically requires that th e concentration of asbest os at the source (worst case) or area of concern (downwind), crosswind, as well a s background (upwind) contribut ions be quantified. See Table 1 (Appendix A) for U.S. EPA/ER T recommended sampling set up for ambient air. Indoor asbestos sampling req uires a different type of strategy which is identified in Table 2 (Appendix A). It i s important to establish background levels o f contaminants in order to develop a reference poin t from which to evaluate the source data. Field blanks and lot blanks can be utilized to determine othe r sources.

Much information can be deri ved from each analytical method previously mentioned. Each analytica l method has specific sampling requirements an d produce results which may or may not be applicabl e to a specific sampling effort. The site samplin g objectives should be carefully id entified so as to select the most appropriate ana lytical method. Additionally, some preparation (i.e., lot blanks results) prior to site sampling may be required, these requirements ar e specified in the analytical methods.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

3.1 Sample Preservation

No preservation is required for asbestos samples.

3.2 Sample Handling, Container an d Storage Procedures

1. Place a sample label on the cassett e indicating a unique sampling number. D o not put sampling cassettes in shirt or coa t pockets as the filter can pick up fibers. The original cassette box is used to hold th e samples.

2. Wrap the cassette individually in a plasti c sample bag. Each bag should be marke d indicating sample identifica tion number, total volume, and date.

3. The wrapped sampling cassettes should b e placed upright in a rigid container so that the cassette cap is on top and cassette base is on bottom. Use enough packing material t o prevent jostling or damage. Do not us e vermiculite as packing material for samples. If possible, hand carry to lab.

4. Provide appropriate documentation wit h samples (i.e., chain of custody and requested analytical methodology).

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Flow rates exceeding 16 liters/minute (L/min) which could result in filter destructi on due to (a) failure of its physical support under force from the increase d pressure drop; (b) leakage of air around the filte r mount so that the filter is bypassed, or (c) damage to the asbestos structures due to increased impac t velocities.

4.1 U.S. EPA's Superfund Method

4.1.1 Direct-transfer TEM Specimen Preparation Methods

Direct-Transfer TEM specimen preparation methods have the following significant interferences:

- The achievable detection limit is restricted by the particulate density on the filter, which in turn is controlled by the sampled air volume and the total suspended particulate concentration in the atmosphere being sampled.
- The precision of the result is dependent on the uniformity of the deposit of asbestos structures on the sample collection filter.
- Air samples must be collected so that they have particulate and fiber loadings within narrow ranges. If too high a particulate loading occurs on the filter, it is not possible to prepare satisfactory TEM specimens by a direct-transfer method. If too high a fiber loading occurs on the filter, even if satisfactory TEM specimens can be prepared, accurate fiber counting will not be possible.

4.1.2 Indirect TEM Specimen Preparation Methods

Indirect TEM specimen preparation methods have the following interferences:

- The size distribution of asbestos structures is modified.
- There is increased opportunity for fiber loss or introduction of extraneous contamination.
- When sample collection filters are ashed, any fiber contamination in the filter medium is concentrated on the TEM specimen grid.

It can be argued that direct methods yield an under-estimate of the asbestos structure concentration because many of the asbestos fibers present are concealed by other particulate material with which they are associated. Conversely, indirect methods can be considered to yield an over-estimate because some types of complex asbestos structures disintegrate during the preparation, resulting in an increase in the numbers of structures counted.

4.2 U.S. EPA's Modified Yamate Method for TEM

High concentrations of background dust interfere with fiber identification.

4.3 NIOSH Method for TEM

Other amphibole particles that have aspect ratios greater than 3:1 and elemental compositions similar to the asbestos minerals may interfere in the TEM analysis. Some non-amphibole minerals may give electron diffraction patterns similar to amphiboles. High concentrations of background dust interfere with fiber identification.

4.4 NIOSH Method for PCM

PCM cannot distinguish asbestos from non-asbestos fibers; therefore, all particles meeting the counting criteria are counted as total asbestos fibers. Fiber less than 0.25 um in length will not be detected by this method. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

5.0 EQUIPMENT/MATERIALS

5.1 Sampling Pump

The constant flow or critical orifice controlled sampling pump should be capable of a flow-rate and pumping time sufficient to achieve the desired volume of air sampled.

The lower flow personal sampling pumps generally provide a flow rate of 20 cubic centimeters/minute (cc/min) to 4 L/min. These pumps are usually battery powered. High flow pumps are utilized when flow rates between 2 L/min to 20 L/min are required. High flow pumps are used for short sampling periods so as to obtain the desired sample volume. High flow pumps usually run on AC power and can be plugged into a nearby outlet. If an outlet is not available then a generator should be obtained. The generator should be positioned downwind from the sampling pump. Additional voltage may be required if more than one pump is plugged into the same generator. Several

electrical extension cord s may be required if sampling locations are remote.

The recommended volume for the Superfund method (Phase I) requires approximately 20 hours to collect. Such pumps typically draw 6 amps at full power s o that 2 lead/acid batteries should provide sufficien t power to collect a full sample. The use of lin e voltage, where available, eliminates the difficultie s associated with transporting stored electrical energy.

A stand should be used to hold th e filter cassette at the desired height for sampling and t he filter cassette shall be isolated from the vibrations of the pump.

5.2 Filter Cassette

The cassettes are purchased wit h the required filters in position, or can be assembled in a laminar flow hood or clean area. When the filters are in position, a shrink cellulose band or adhesive tape should b e applied to cassette joints to prevent air leakage.

5.2.1 TEM Cassette Requirements

Commercially available field monitors, comprisin g 25 mm diameter three-piece cassettes, wit h conductive extension cowls shall be used for sample collection. The cassette must be new and no t previously used. The cassette shall be loaded with an MCE filter of pore size 0.45 µm, and supplied from a lot number which has been qualified as lo w background for asbestos determination. The cowl s should be constructed of electrically conductin g material to minimize electrostatic effects. The filte r shall be backed by a 5 µm pore size MCE filte r (Figure 1, Appendix B).

5.2.2 PCM Cassette Requirements

NIOSH Method 7400, PCM involves using a 0.8 t o 1.2 µm mixed cellulose ester membrane, 25 m m diameter, 50 mm conductive cowl on cassette (Figure 2, Appendix B). Some labs are able to perform PCM and TEM analysis on the same filter; however, thi s should be discussed with the laboratory prior t o sampling.

5.3 Other Equipment

- Inert tubing with glass cyclone and hose barb
- Whirlbags (plastic bags) for cassettes
- Tools - small screw drivers
- Container - to keep samples upright
- Generator or electrical outlet (may not b e required)
- Extension cords (may not be required)
- Multiple plug outlet
- Sample labels
- Air data sheets
- Chain of Custody records

6.0 REAGENTS

Reagents are not required for the preservation o f asbestos samples.

7.0 PROCEDURES

7.1 Air Volumes and Flow Rates

Sampling volumes are deter mined on the basis of how many fibers need to be collected for reliabl e measurements. Therefore, one must estimate ho w many airborne fibers m ay be in the sampling location.

Since the concentration of airborne aeroso l contaminants will have some effect on the sample, the following is a suggested criteria to assist in selecting a flow rate based on rea l-time aerosol monitor (RAM) readings in milligrams/cubic meter (mg/m ^{3}).

	Concentration	Flow Rate
• Low RAM readings:	<6.0 mg/m ^{3}	11-15. L/min
• Medium RAM readings:	>6.0 mg/m ^{3}	7.5 L/min
• High RAM readings:	>10. mg/m ^{3}	2.5 L/min

In practice, pumps that are availab le for environmental sampling at remote locations operate under a maximum load of approximately 12 L/min.

7.1.1 U.S. EPA's Superfund Method

The Superfund Method incorporates an indirec t preparation procedure to provide flexibility in th e amount of deposit that be can be tolerated on th e sample filter and to allow for the selectiv e concentration of asbestos prior to analysis. T o minimize contributions to background contamination from asbestos present in the plastic matrices o f membrane filters while allowing for sufficien t quantities of asbestos to be collected, this method also requires the collection of a larger volume of air pe r unit area of fi lter than has traditionally been collected

for asbestos analysis. Due to the need to collect large volumes of air, higher sampling flow rates are recommended in this method than have generally been employed for asbestos sampling in the past. As an alternative, samples may be collected over longer time intervals. However, this restricts the flexibility required to allow samples to be collected while uniform meteorological conditions prevail.

The sampling rate and the period of sampling should be selected to yield as high a sampled volume as possible, which will minimize the influence of filter contamination. Wherever possible, a volume of 15 cubic meters (15,000 L) shall be sampled for those samples intended for analysis only by the indirect TEM preparation method (Phase 1 samples). For those samples to be prepared by both the indirect and the direct specimen preparation methods (Phase 2 samples), the volumes must be adjusted so as to provide a suitably-loaded filter for the direct TEM preparation method. One option is to collect filters at several loadings to bracket the estimated optimum loading for a particular site. Such filters can be screened in the laboratory so that only those filters closest to optimal loading are analyzed. It has been found that the volume cannot normally exceed 5 cubic meters (5000 L) in an urban or agricultural area, and 10 cubic meters (10,000 L) in a rural area for samples collected on a 25 mm filter and prepared by a direct-transfer technique.

An upper limit to the range of acceptable flow rates for this method is 15 L/min. At many locations, wind patterns exhibit strong diurnal variations. Therefore, intermittent sampling (sampling over a fixed time interval repeated over several days) may be necessary to accumulate 20 hours of sampling time over constant wind conditions. Other sampling objectives also may necessitate intermittent sampling. The objective is to design a sampling schedule so that samples are collected under uniform conditions throughout the sampling interval. This method provides for such options. Air volumes collected on Phase I samples are maximized (<16 L/min). Air volumes collected on Phase 2 samples are limited to provide optimum loading for filters to be prepared by a direct-transfer procedure.

7.1.2 U.S. EPA's Modified Yamate Method for TEM

U.S. EPA's TEM method requires a minimum volume of 560 L and a maximum volume of 3,800 L in order to obtain an analytical sensitivity of 0.005 structures/cc. The optimal volume for TEM is 1200 L to 1800 L. These volumes are determined using a 200 mesh EM grid opening with a 25-mm filter cassette. Changes in volume would be necessary if a 37-mm filter cassette is used since the effective area of a 25 mm (385 sq mm) and 37 mm (855 sq m) differ.

7.1.3 NIOSH Method for TEM and PCM

The minimum recommended volume for TEM and PCM is 400 L at 0.1 fiber/cc. Sampling time is adjusted to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for eight hours (700 to 2800 L) is appropriate in non-dusty atmospheres containing 0.1 fiber/cc. Dusty atmospheres i.e., areas with high levels of asbestos, require smaller sample volumes (<400 L) to obtain countable samples.

In such cases, take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3,000 to 10,000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If > 50% of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration. Do not exceed 0.5 mg total dust loading on the filter.

7.2 Calibration Procedures

In order to determine if a sampling pump is measuring the flow rate or volume of air correctly, it is necessary to calibrate the instrument. Sampling pumps should be calibrated immediately before and after each use. Preliminary calibration should be conducted using a primary calibrator such as a soap bubble type calibrator, (e.g., a Buck Calibrator, Gilibrator, or equivalent primary calibrator) with a representative filter cassette installed between the pump and the calibrator. The representative sampling cassette can be reused for calibrating other pumps that will be used for asbestos sampling. The same cassette lot used for sampling should also be used for the calibration. A sticker should be affixed to the outside of the extension cowl marked "Calibration Cassette."

A rotameter can be used provided it has been recently precalibrated with a primary calibrator. Three separate constant flow calibration readings should be obtained both before sampling and after sampling. Should the flow rate change by more than 5% during the sampling period, the average of the pre- and post-calibration rates will be used to calculate the total sample volume. The sampling pump used shall provide a non-fluctuating air-flow through the filter, and shall maintain the initial volume flow-rate to within ± 10% throughout the sampling period. The mean value of these flow-rate measurements shall be used to calculate the total air volume sampled. A constant flow or critical orifice controlled pump meets these requirements. If at any time the measurement indicates that the flow-rate has decreased by more than 30%, the sampling shall be terminated. Flexible tubing is used to connect the filter cassette to the sampling pump. Sampling pumps can be calibrated prior to coming on-site so that time is saved when performing on-site calibration.

7.2.1 Calibrating a Personal Sampling Pump with an Electronic Calibrator

1. See Manufacturer's manual for operational instructions.

2. Set up the calibration train as shown in (Figure 3, Appendix B) using a sampling pump, electronic calibrator, and a representative filter cassette. The same lot sampling cassette used for sampling should also be used for calibrating.

3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 foot) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the electronic calibrator.

4. Turn the electronic calibrator and sampling pump on. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

5. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

6. Perform the calibration three times until the desired flow rate of ± 5% is attained.

7.2.2 Calibrating a Rotameter with an Electronic Calibrator

1. See manufacturer's manual for operational instructions.

2. Set up the calibration train as shown in (Figure 4, Appendix B) using a sampling pump, rotameter, and electronic calibrator.

3. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6 ° vertical.

4. Turn the electronic calibrator and sampling pump on.

5. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

6. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

7. Record the electronic calibrator flow rate reading and the corresponding rotameter reading. Indicate these values on the rotameter (sticker). The rotameter should be able to work within the desired flow range. Readings can also be calibrated for 10 cm^3 increments for Low Flow rotameters, 500 cm^3 increments for medium flow rotameters and 1 liter increments for high flow rotameters.

8. Perform the calibration three times until the desired flow rate of ± 5% is attained. Once on site, a secondary calibrator, i.e., rotameter may be used to calibrate sampling pumps.

7.2.3 Calibrating a Personal Sampling Pump with a Rotameter

1. See manufacturer's manual for Rotameter's Operational Instructions.

2. Set up the calibration train as shown in (Figure 5, Appendix B) using a rotameter, sampling pump, and a representative sampling cassette.

3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 ft) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the rotameter.

4. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within 6° vertical.

5. Turn the sampling pump on.

6. Turn the flow adjust screw (or knob) on the personal sampling pump until the float ball on the rotameter is lined up with the precalibrated flow rate value. A sticker on the rotameter should indicate this value.

7. A verification of calibration is generally performed on-site in the clean zone immediately prior to the sampling.

7.3. Meteorology

It is recommended that a meteorological station be established. If possible, sample after two to three days of dry weather and when the wind conditions are at 10 mph or greater. Record wind speed, wind direction, temperature, and pressure in a field logbook. Wind direction is particularly important when monitoring for asbestos downwind from a fixed source.

7.4 Ambient Sampling Procedures

7.4.1 Pre-site Sampling Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.

2. Obtain necessary sampling equipment and ensure it is in working order and fully charged (if necessary).

3. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety plan.

4. Once on-site the calibration is performed in the clean zone. The calibration procedures are listed in Section 7.2.

5. After calibrating the sampling pump, mobilize to the sampling location.

7.4.2 Site Sampling

1. To set up the sampling train, attach the air intake hose to the cassette base. Remove the cassette cap (Figure 6 and 7, Appendix B). The cassette should be positioned downward, perpendicular to the wind

2. If AC or DC electricity is required then turn it on. If used, the generator should be placed 10 ft. downwind from the sampling pump.

3. Record the following in a field logbook: date, time, location, sample identification number, pump number, flow rate, and cumulative time.

4. Turn the pump on. Should intermittent sampling be required, sampling filters must be covered between active periods of sampling. To cover the sample filter: turn the cassette to face upward, place the cassette cap on the cassette, remove the inlet plug from the cassette cap, attach a rotameter to the inlet opening of the cassette cap to measure the flow rate, turn off the sampling pump, place the inlet plug into the inlet opening on the cassette cap. To resume sampling: remove the inlet plug, turn on the sampling pump, attach a rotameter to measure the flow rate, remove the cassette cap, replace the inlet plug in the cassette cap and invert the cassette, face downward and perpendicular to the wind.

5. Check the pump at sampling midpoint if sampling is longer than 4 hours. The generators may need to be regased depending on tank size. If a filter darkens in appearance or if loose dust is seen in the filter, a second sample should be started.

6. At the end of the sampling period, orient the cassette up, turn the pump off.

7. Check the flow rate as shown in Sectio n 7.2.3. When sampling open-faced, th e sampling cap should be replaced before post calibrating. Use the same cassette used fo r sampling for post calibration (increas e dust/fiber loading may have altered the flow rate.

8. Record the post flow rate.

9. Record the cumulative time or run.

10. Remove the tubing from the samplin g cassette. Still holding the cassette upright , replace the inlet plug on the cassette cap and the outlet plug on the cassette base.

7.4.3. Post Site Sampling

1. Follow handling procedures in Section 3.2 , steps 1-4.

2. Obtain an electronic or hard copy o f meteorological data which occurred durin g the sampling event. Record weather: win d speed, ambient temperature, wind direction, and precipitation. Obtaining weather dat a several days prior to the sampling event can also be useful.

7.5 Indoor Sampling Procedures

PCM analysis is used for indoor air samples. Whe n analysis shows total fiber count above the OSH A action level 0.1 f/cc then TEM (U.S. EPA's Modified Yamate Method) is used to identify asbestos fro m non-asbestos fibers.

Samplin g pumps should be placed four to five fee t above ground level away from obstructions that may influence air flow. The pump can be placed on a table or counter. Refer to Table 2 (Appendix A) for a summary of indoor sampling locations and rational e for selection.

Indoor sampling utilizes high flow rates to increased sample volumes (2000 L for PCM and 2800 to 4200 L for TEM) in order to obtain lower detection limit s below the standard, (i.e., 0.01 f/cc or lower [PCM] and 0.005 structures/cc or lower [TEM]).

7.5.1 Aggressive Sampling Procedures

Sampling equipment at fixed locations may fail t o detect the presence of asbestos fibers. Due to limited air movement, many fibers may settle out of the ai r onto the floor and other surfaces and may not b e captured on the fil ter. In the past, an 8-hour sampling period was recommended to cover various ai r circulation conditions. A quicker and more effective way to capture asbestos fibers is to circulate the ai r artificially so that the fibers remain airborne durin g sampling. The results from this sampling optio n typifies worst case condition. This is referred to a s aggressive air sampling for asbestos. Refer to Table 2 for sample station locations.

1. Before starting the sampling pumps, direc t forced air (such as a 1-horsepower lea f blower or large fan) against walls, ceilings , floors, ledges, and other surfaces in the room to initially dislodge fibers from surfaces . This should take at least 5 minutes per 1000 sq. ft. of floor.

2. Place a 20-inch fan in the center of the room. (Use one fan per 10,000 cubic feet of roo m space.) Place the fan on slow speed an d point it toward the ceiling.

3. Follow procedur es in Section 7.4.1 and 7.4.2 (Turn off the pump and then the fan(s) when sampling is complete.).

4. Follow handling procedures in Section 3.2 , steps 1-4.

8.0 CALCULATIONS

The sample volume is calculated from the averag e flow rate of the pump multiplied by the number o f minutes the pump w as running (volume = flow rate X time in minutes). The sample volume should b e submitted to the laborato ry and identified on the chain of custody for each sample (zero for lot, field and trip blanks).

The concentration result is calculated using th e sample volume and the number s of asbestos structures reported after the ap plication of the cluster and matrix counting criteria.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

Follow all QA/QC req uirements from the laboratories as well as the analytical methods.

9.1 TEM Requirements

1. Examine lot blanks to determine th e background asbes tos structure concentration.

2. Examine field blanks to determine whethe r there is contamination by extraneou s asbestos structures during specime n preparation.

3. Examine of labor atory blanks to determine if contamination is being introduced durin g critical phases of the laboratory program.

4. To determine if the laboratory ca n satisfactorily analyze samples of know n asbestos structure concentrations, referenc e filters shall be examined. Reference filter s should be maintained as part of th e laboratory's Quality Assurance program.

5. To minimize subjective effects, som e specimens should be r ecounted by a different microscopist.

6. Asbestos laboratories shall be accredited by the National Voluntary Laborator y Accreditation Program.

7. At this time, performance evaluation samples for asbestos in air are not available fo r Removal Program Activities.

9.2 PCM Requirements

1. Examine reference slides of know n concentration to determine the analyst' s ability to satisfactorily count fibers . Reference slides should be maintained a s part of the laboratory's quality assuranc e program.

2. Examine fi eld blanks to determine if there is contamination by extraneous structure s during sample handling.

3. Some samples should be relabeled the n submitted for counting by th e same analyst to determine possible bias by the analyst.

4. Participation in a profic iency testing program such as the AIHA-NIOSH proficienc y analytical testing (PAT) program.

10.0 DATA VALIDATION

Results of quality control samples will be evaluate d for contamination. This information will be utilize d to qualify the environmental sample result s accordingly with the project's data quality objectives.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials , follow U.S. EPA, OSHA, and corporate health an d safety procedures. More specifically, when entering an unknown situation involving asbestos, a powere d air purifying respirator (PAPR) (full face-piece) i s necessary in co njunction with HEPA filter cartridges. See applicable regulations for action level, PEL, TLV, etc. If previous sampling indicates asbesto s concentrations are below personal health and safet y levels, then Level D personal protection is adequate.

12.0 REFERENCES

(1) Environmental Asbestos Assessmen t Manual, Superfund Method for th e Determination of Asbestos in Ambient Air , Part 1: Method, EPA/540/2-90/005a, Ma y 1990, and Part 2: Technical Backgroun d Document, EPA/540/2-90/005b, May 1990.

(2) Methodology for the Measurement o f Airborne Asbestos by Electron Microscopy, EPA's Report No. 68-02-3266, 1984, G . Yamate, S.C. Agarwal, and R. D. Gibbons.

(3) National Institute for Occupational Safet y and Health. NIOSH Manual of Analytica l Method. Third Edition. 1987.

(4) U.S. Environmental Protection Agency . Code of Federal Regulations 40 CFR 763 . July 1, 1987. Code of Federal Regulation s 40 CFR 763 Addendum. October 30, 1987.

(5) U.S. Environmental Protection Agency . Asbestos-Containing Materials in Schools ; Final Rule and Notice. 52 FR 41826.

(6) Occupational Safety and Healt h Administration. Code of Fede ral Regulations 29 CFR 1910.1001. Washington, D.C . 1987.

APPENDIX A

Tables

TABLE 1.

SAMPLE STATIONS FOR OUTDOOR SAMPLING

Sample Station Location	Sample Numbers	Rationale
Upwind/Background [1]	Collect a minimum of two simultaneous upwind/background samples 30 ° apart from the prevailing windlines.	Establishes background fiber levels.
Downwind	Deploy a minimum of 3 sampling stations in a 180 degree arc downwind from the source.	Indicates if asbestos is leaving the site.
Site Representative and/or Worst Case	Obtain one site representative sample which shows average condition on-site or obtain worst case sample (optional).	Verify and continually confirm and document selection of proper levels of worker protection.

[1] More than one background station may be required if the asbestos originates from different sources.

APPENDIX A (Cont'd)

Tables

TABLE 2

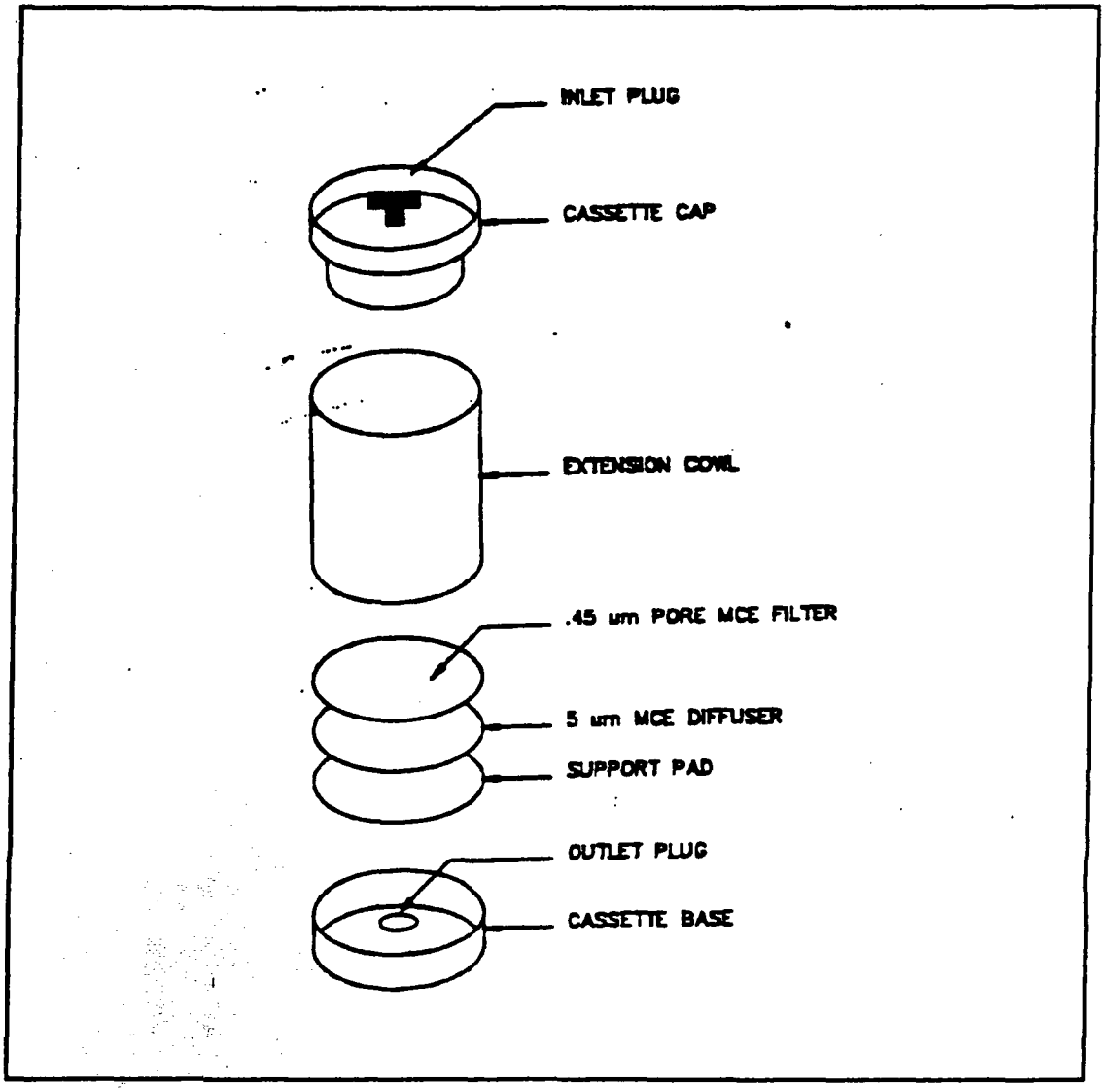
SAMPLE STATIONS FOR INDOOR SAMPLING

Sample Station Location	Sample Numbers	Rationale
Indoor Sampling	If a work site is a single room, disperse 5 samplers throughout the room. If the work site contains up to 5 rooms, place at least one sampler in each room. If the work site contains more than 5 rooms, select a representative sample of the rooms.	Establishes representative samples from a homogeneous area.
Upwind/Background	If outside sources are suspected, deploy a minimum of two simultaneous upwind/background samples 30 ° apart from the prevailing windlines.	Establish whether indoor asbestos concentrations are coming from an outside source.
Worst Case	Obtain one worst case sample, i.e., aggressive sampling (optional).	Verify and continually confirm and document selection of proper levels of worker protection.

APPENDIX B

Figures

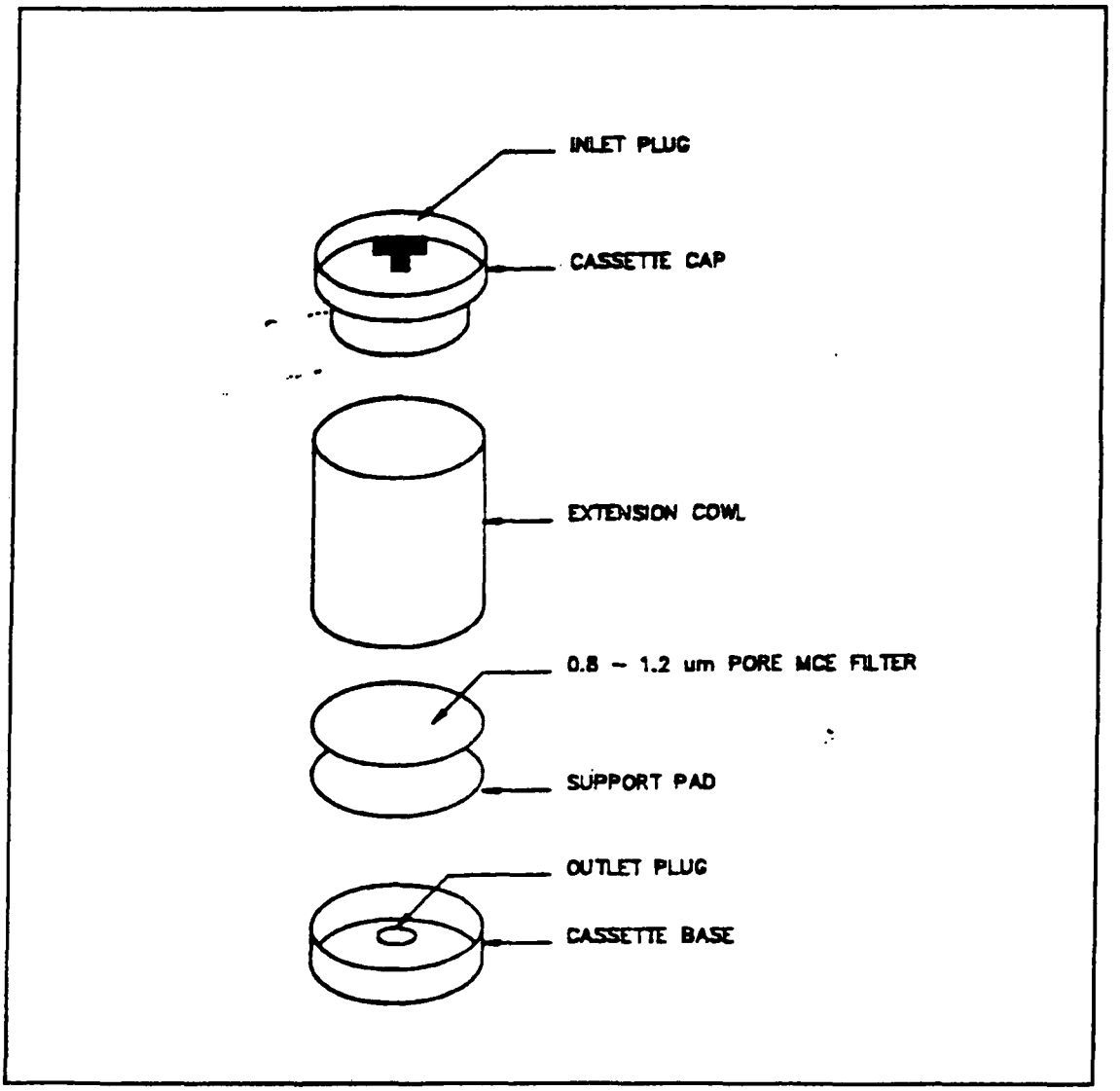
FIGURE 1. *Transmission Electron Microscopy Filter Cassette*



APPENDIX B (Cont'd)

Figures

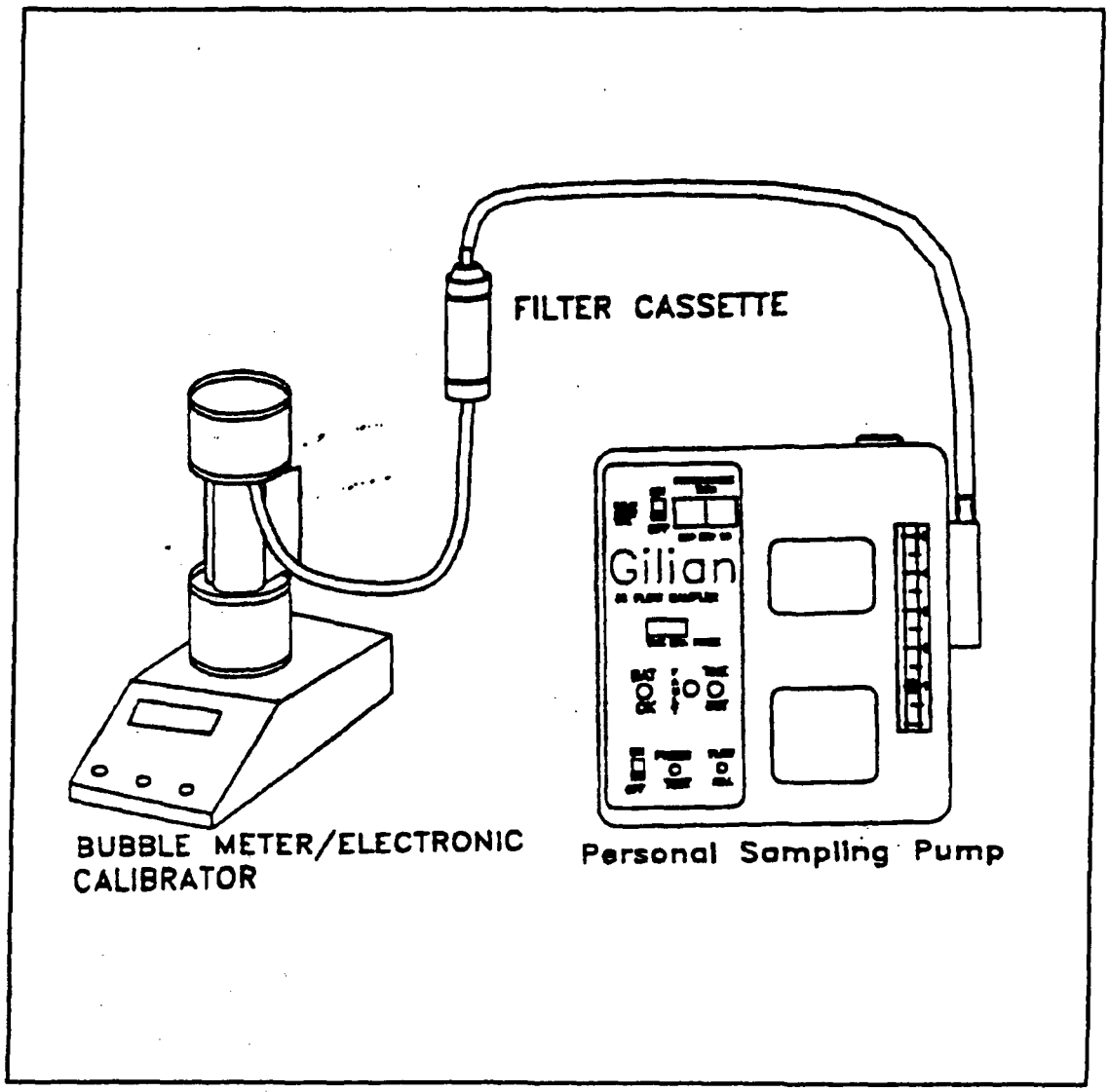
FIGURE 2. Phase Contrast Microscopy Filter Cassette



APPENDIX B (Cont'd)

Figures

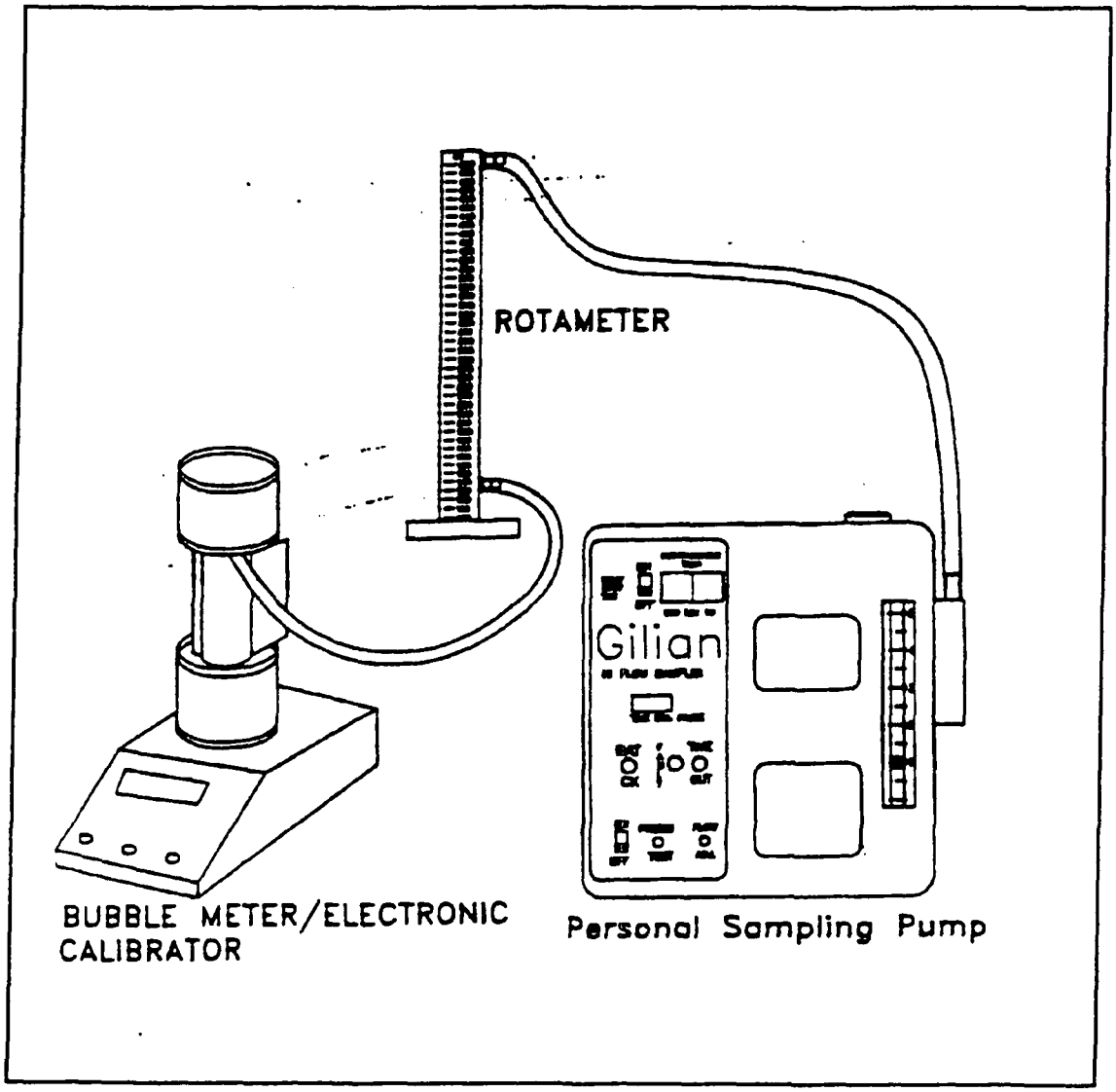
FIGURE 3. Calibrating a Personal Sampling Pump with a Bubble Meter



APPENDIX B (Cont'd)

Figures

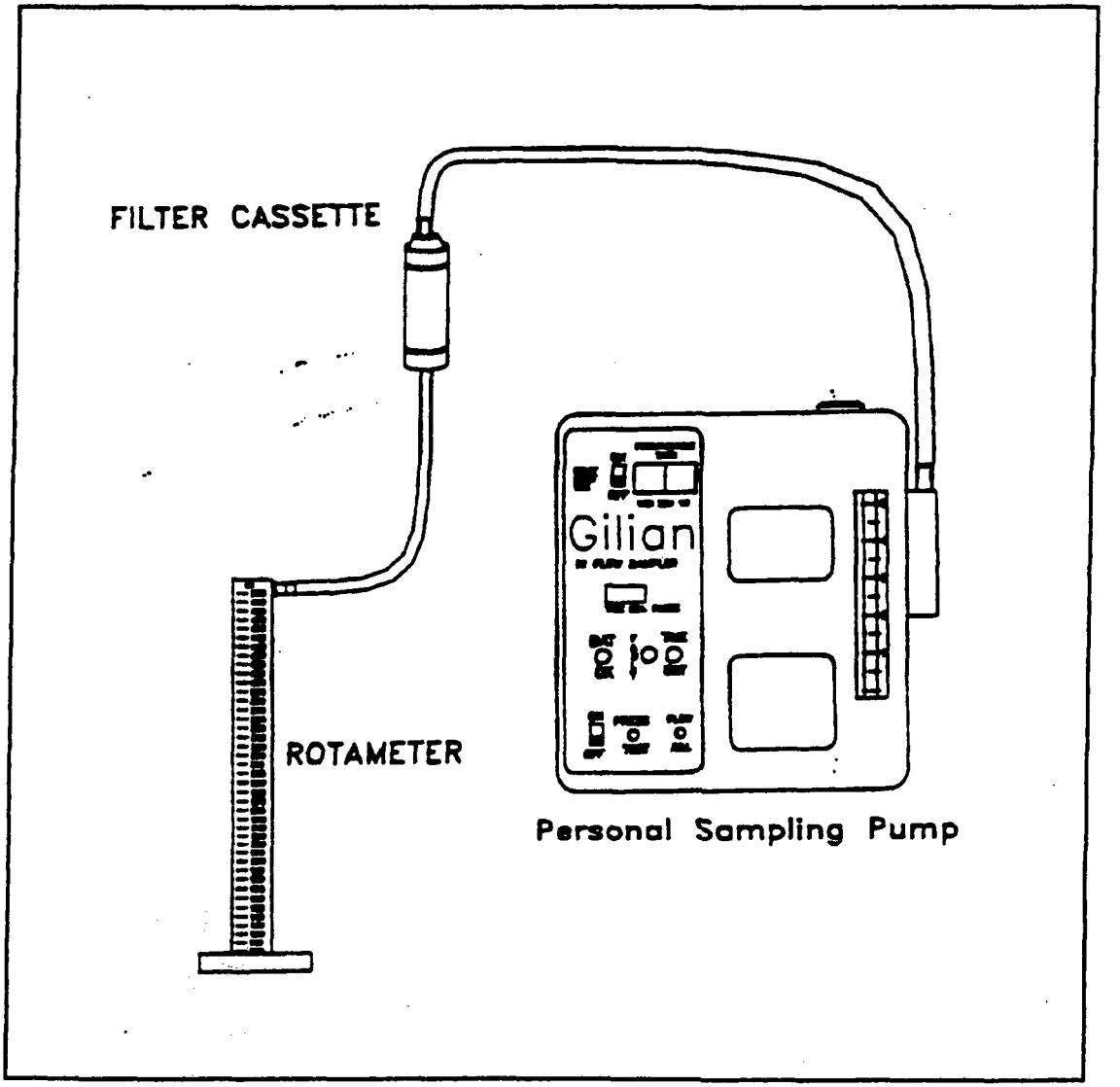
FIGURE 4. Calibrating a Rotameter with a Bubble Meter



APPENDIX B (Cont'd)

Figures

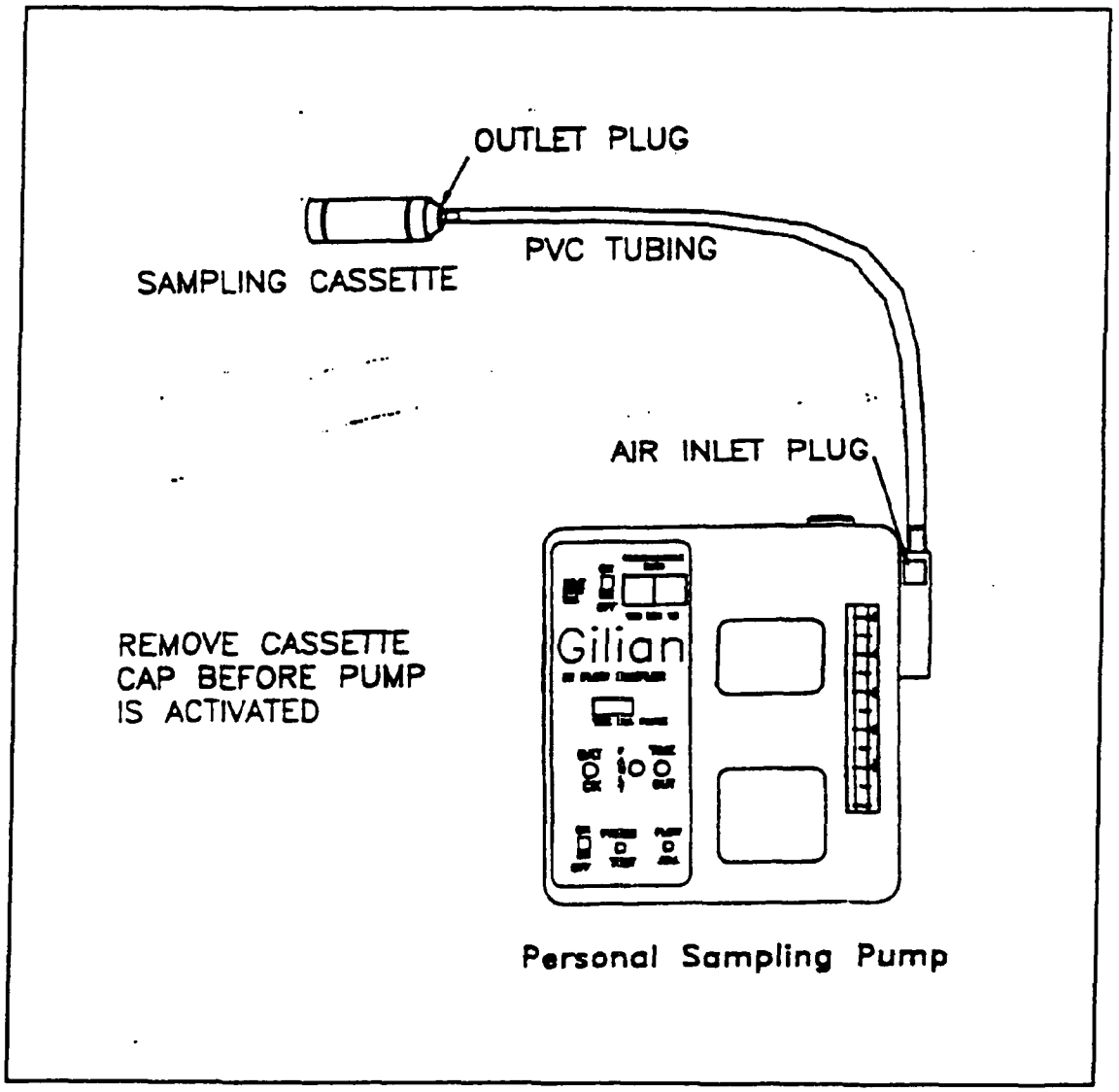
FIGURE 5. Calibrating a Sampling Pump with a Rotameter



APPENDIX B (Cont'd)

Figures

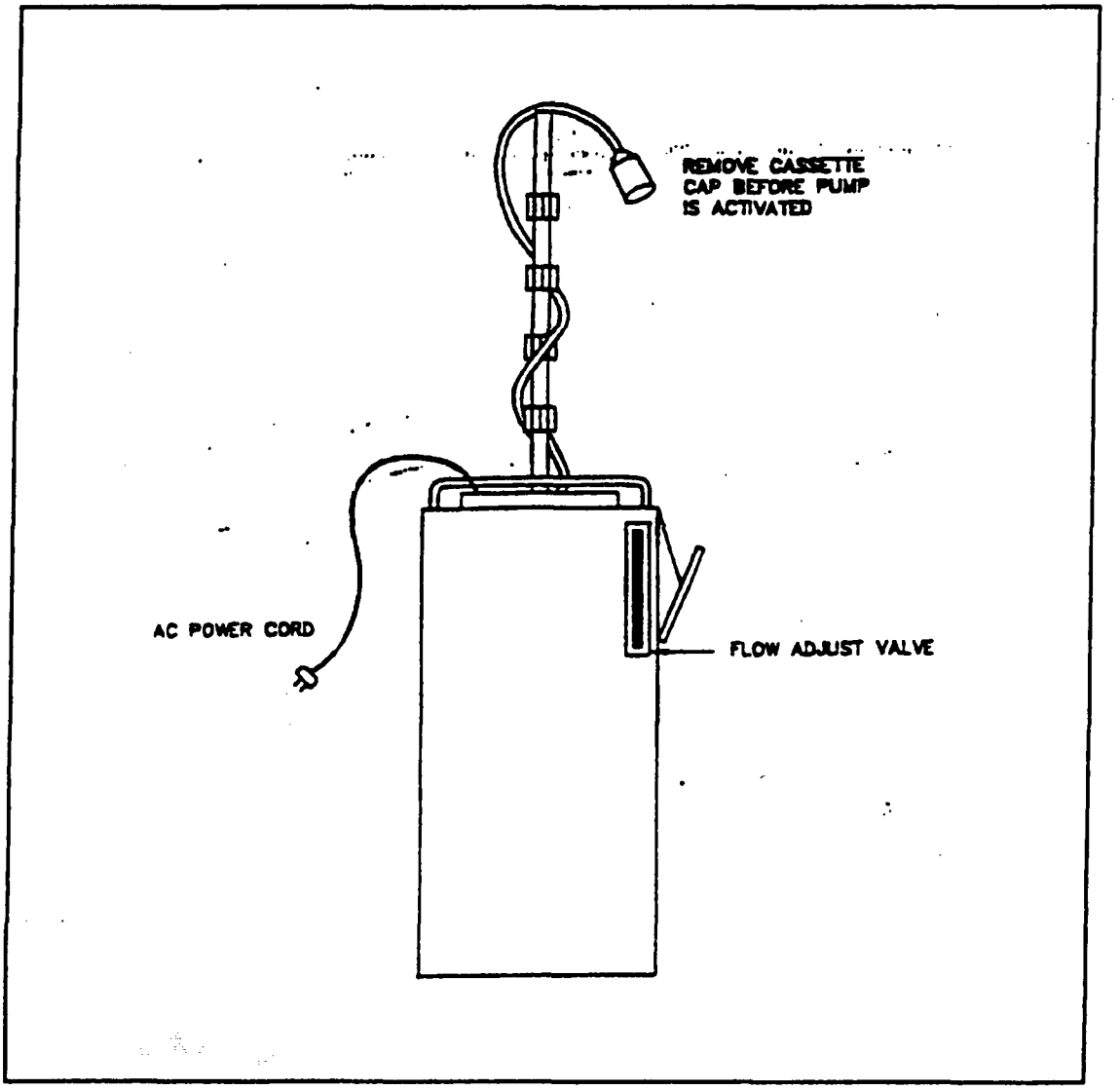
FIGURE 6. Personal Sampling Train for Asbestos



APPENDIX B (Cont'd)

Figures

FIGURE 7. High Flow Sampling Train for Asbestos



Appendix C
Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations. ASTM D-5755-95

Designation: D 5755 – 95

AMERICAN SOCIETY FOR TESTING AND MATERIALS
1916 Race St. Philadelphia, Pa 19103
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If not listed in the current combined index, will appear in the next edition.

Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations[1]

This standard is issued under the fixed designation D 5755; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure to (*a*) identify asbestos in dust and (*b*) provide an estimate of the concentration of asbestos in the sampled dust reported as the number of asbestos structures per unit area of sampled surface.

1.1.1 If an estimate of the asbestos mass is to be determined, the user is referred to Test Method D 5756.

1.2 This test method describes the equipment and procedures necessary for sampling, by a microvacuum technique, non-airborne dust for levels of asbestos structures. The non-airborne sample is collected inside a standard filter membrane cassette from the sampling of a surface area for dust which may contain asbestos.

1.2.1 This procedure uses a microvacuuming sampling technique. The collection efficiency of this technique is unknown and will vary among substrates. Properties influencing collection efficiency include surface texture, adhesiveness, electrostatic properties and other factors.

1.3 Asbestos identified by transmission electron microscopy (TEM) is based on morphology, selected area electron diffraction (SAED), and energy dispersive X-ray analysis (EDXA). Some information about structure size is also determined.

1.4 This test method is generally applicable for an estimate of the concentration of asbestos structures starting from approximately 1000 asbestos structures per square centimetre.

1.4.1 The procedure outlined in this test method employs an indirect sample preparation technique. It is intended to disperse aggregated asbestos into fundamental fibrils, fiber bundles, clusters, or matrices that can be more accurately quantified by transmission electron microscopy. However, as with all indirect sample preparation techniques, the asbestos observed for quantification may not represent the physical form of the asbestos as sampled. More specifically, the procedure described neither creates nor destroys asbestos, but it may alter the physical form of the mineral fibers.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 1739 Test Method for the Collection and Measurement of Dustfall (Settleable Particulate Matter)[3]

D 3195 Practice for Rotameter Calibration[3]

D 3670 Guide for Determination of Precision and Bias of Methods of Committee D-22[3]

D 5756 Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Concentration[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *asbestiform*—a special type of fibrous habit in which the fibers are separable into thinner fibers and ultimately into fibrils. This habit accounts for greater flexibility and higher tensile strength than other habits of the same mineral. For more information on asbestiform mineralogy, see Refs (1),[4] (2) and (3).

3.1.2 *asbestos*—a collective term that describes a group of naturally occurring, inorganic, highly fibrous, silicate dominated minerals, which are easily separated into long, thin, flexible fibers when crushed or processed.

DISCUSSION—Included in the definition are the asbestiform varieties of: serpentine (chrysotile); riebeckite (crocidolite); grunerite (grunerite asbestos); anthophyllite (anthophyllite asbestos); tremolite (tremolite asbestos); and actinolite (actinolite asbestos). The amphibole mineral compositions are defined according to nomenclature of the International Mineralogical Association (3).

Asbestos	Chemical Abstract Service No.[5]
Chrysotile	12001-29-5
Crocidolite	12001-28-4
Grunerite Asbestos	12172-73-5
Anthophyllite Asbestos	77536-67-5
Tremolite Asbestos	77536-68-6
Actinolite Asbestos	77536-66-4

3.1.3 *fibril*—a single fiber that cannot be separated into

[1] This test method is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.07 on Sampling and Analysis of Asbestos.

Current edition approved August 15, 1995. Published October 1995.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.03.

[4] The boldface numbers in parentheses refer to the list of references at the end of this test method.

[5] The non-asbestiform variations of the minerals indicated in 3.1.3 have different Chemical Abstract Service (CAS) numbers.

smaller components without losing its fibrous properties or appearance.

3.2 *Descriptions of Terms Specific to This Standard:*

3.2.1 *aspect ratio*—the ratio of the length of a fibrous particle to its average width.

3.2.2 *bundle*—a structure composed of three or more fibers in a parallel arrangement with the fibers closer than one fiber diameter to each other.

3.2.3 *cluster*—a structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings of fibers must have more than two points touching.

3.2.4 *debris*—materials that are of an amount and size (particles greater than 1 mm in diameter) that can be visually identified as to their source.

3.2.5 *dust*—any material composed of particles in a size range of ≤1 mm and large enough to settle by virtue of their weight from the ambient air (see definition for settleable particulate matter in Test Method D 1739).

3.2.6 *fiber*—a structure having a minimum length of 0.5 µm, an aspect ratio of 5:1 or greater, and substantially parallel sides (4).

3.2.7 *fibrous*—of a mineral composed of parallel, radiating, or interlaced aggregates of fibers, from which the fibers are sometimes separable. That is, the crystalline aggregate may be referred to as fibrous even if it is not composed of separable fibers, but has that distinct appearance. The term fibrous is used in a general mineralogical way to describe aggregates of grains that crystallize in a needle-like habit and appear to be composed of fibers. Fibrous has a much more general meaning than asbestos. While it is correct that all asbestos minerals are fibrous, not all minerals having fibrous habits are asbestos.

3.2.8 *indirect preparation*—a method in which a sample passes through one or more intermediate steps prior to final filtration.

3.2.9 *matrix*—a structure in which one or more fibers, or fiber bundles that are touching, are attached to, or partially concealed by a single particle or connected group of non-fibrous particles. The exposed fiber must meet the fiber definition (see 3.2.6).

3.2.10 *structures*—a term that is used to categorize all the types of asbestos particles which are recorded during the analysis (such as fibers, bundles, clusters, and matrices). Final results of the test are always expressed in asbestos structures per square centimetre.

4. Summary of Test Method

4.1 The sample is collected by vacuuming a known surface area with a standard 25 or 37 mm air sampling cassette using a plastic tube that is attached to the inlet orifice which acts as a nozzle. The sample is transferred from inside the cassette to an aqueous solution of known volume. Aliquots of the suspension are then filtered through a membrane. A section of the membrane is prepared and transferred to a TEM grid using the direct transfer method. The asbestiform structures are identified, sized, and counted by TEM, using SAED and EDXA at a magnification of 15 000 to 20 000X.

5. Significance and Use

5.1 This microvacuum sampling and indirect analysis method is used for the general testing of non-airborne dust samples for asbestos. It is used to assist in the evaluation of dust that may be found on surfaces in buildings such as ceiling tiles, shelving, electrical components, duct work, carpet, etc. This test method provides an index of the concentration of asbestos structures in the dust per unit area analyzed as derived from a quantitative TEM analysis.

5.1.1 This test method does not describe procedures or techniques required to evaluate the safety or habitability of buildings with asbestos-containing materials, or compliance with federal, state, or local regulations or statutes. It is the user's responsibility to make these determinations.

5.1.2 At present, a single direct relationship between asbestos-containing dust and potential human exposure does not exist. Accordingly, the user should consider these data in relationship to other available information in their evaluation.

5.2 This test method uses the definition, settleable particulate material, found in Test Method D 1739 as the definition of dust. This definition accepts all particles small enough to pass through a 1 mm (No. 18) screen. Thus, a single, large asbestos containing particle(s) (from the large end of the particle size distribution) dispersed during sample preparation may result in anomalously large asbestos concentration results in the TEM analyses of that sample. It is, therefore, recommended that multiple independent samples are secured from the same area, and a minimum of three samples analyzed by the entire procedure.

6. Interferences

6.1 The following minerals have properties (that is, chemical or crystalline structure) which are very similar to asbestos minerals and may interfere with the analysis by causing a false positive to be recorded during the test. Therefore, literature references for these materials must be maintained in the laboratory for comparison to asbestos minerals so that they are not misidentified as asbestos minerals.

6.1.1 *Antigorite.*

6.1.2 *Palygorskite (Attapulgite).*

6.1.3 *Halloysite.*

6.1.4 *Pyroxenes.*

6.1.5 *Sepiolite.*

6.1.6 *Vermiculite scrolls.*

6.1.7 *Fibrous talc.*

6.1.8 Hornblende and other amphiboles other than those listed in 3.1.2.

6.2 Collecting any dust particles greater than 1 mm in size in this test method may cause an interference and, therefore, must be avoided.

7. Materials and Equipment

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without

lessening the accuracy of the determination.[6]

7.2 *Transmission Electron Microscope (TEM)*, an 80 to 120 kV TEM, capable of performing electron diffraction, with a fluorescent screen inscribed with calibrated gradations, is required. The TEM must be equipped with energy dispersive X-ray spectroscopy (EDXA) and it must have a scanning transmission electron microscopy (STEM) attachment or be capable of producing a spot size of less than 250 nm in diameter in crossover.

7.3 *Energy Dispersive X-ray System (EDXA).*

7.4 *High Vacuum Carbon Evaporator*, with rotating stage.

7.5 *High Efficiency Particulate Air (HEPA)*, filtered negative flow hood.

7.6 *Exhaust or Fume Hood.*

7.7 *Particle-free Water* (ASTM Type II, see Specification D 1193).

7.8 *Glass Beakers* (50 mL).

7.9 *Glass Sample Containers*, with wide mouth screw cap (200 mL) or equivalent sealable container (height of the glass sample container should be approximately 13 cm high by 6 cm wide).

7.10 *Waterproof Markers.*

7.11 *Forceps* (tweezers).

7.12 *Ultrasonic Bath*, table top model (100 W).

7.13 *Graduated Pipettes* (1, 5, 10 mL sizes), glass or plastic.

7.14 *Filter Funnel*, either 25 mm or 47 mm, glass or disposable. Filter funnel assemblies, either glass or disposable plastic, and using either a 25 mm or 47 mm diameter filter.

7.15 *Side Arm Filter Flask*, 1000 mL.

7.16 *Mixed Cellulose Ester (MCE) Membrane Filters*, 25 or 47 mm diameter, ≤0.22 µm and 5 µm pore size.

7.17 *Polycarbonate (PC) Filters*, 25 or 47 mm diameter, ≤0.2 µm pore size.

7.18 *Storage Containers*, for the 25 or 47 mm filters (for archiving).

7.19 *Glass Slides*, approximately 76 by 25 mm in size.

7.20 *Scalpel Blades*, No. 10, or equivalent.

7.21 *Cabinet-type Desiccator*, or low temperature drying oven.

7.22 *Chloroform*, reagent grade.

7.23 *Acetone*, reagent grade.

7.24 *Dimethylformamide (DMF).*

7.25 *Glacial Acetic Acid.*

7.26 *1-methyl-2-pyrrolidone.*

7.27 *Plasma Asher*, low temperature.

7.28 *pH Paper.*

7.29 *Air Sampling Pump*, low volume personal-type, capable of achieving a flow rate of 1 to 5 L/min.

7.30 *Rotameter.*

7.31 *Air Sampling Cassettes*, 25 mm or 37 mm, containing 0.8 µm or smaller pore size MCE or PC filters.

7.32 *Cork Borer*, 7 mm.

7.33 *Non-Asbestos Mineral*, references as outlined in 6.1.

7.34 *Asbestos Standards*, as outlined in 3.1.2.

7.35 *Tygon*[7] *Tubing*, or equivalent.

7.36 *Small Vacuum Pump*, that can maintain a pressure of 92 kPa.

7.37 *Petri Dishes*, large glass, approximately 90 mm in diameter.

7.38 *Jaffe Washer*, stainless steel or aluminum mesh screen, 30 to 40 mesh, and approximately 75 mm by 50 mm in size.

7.39 *Copper TEM Finder Grids*, 200 mesh.

7.40 *Carbon Evaporator Rods.*

7.41 *Lens Tissue.*

7.42 *Ashless Filter Paper Filters*, 90 mm diameter.

7.43 *Gummed Paper Reinforcement Rings.*

7.44 *Wash Bottles*, plastic.

7.45 *Reagent Alcohol*, HPLC Grade (Fisher A995 or equivalent).

7.46 *Opening Mesh Screen*, plastic, 1.0 by 1.0 mm, (Spectra-Mesh #146410 or equivalent).

7.47 *Diffraction Grating Replica.*

8. Sampling Procedure for Microvacuum Technique

8.1 For sampling asbestos-containing dust in either indoor or outdoor environments, commercially available cassettes must be used. Air monitoring cassettes containing 25 mm or 37 mm diameter mixed cellulose ester (MCE) or polycarbonate (PC) filter membranes with a pore size less than or equal to 0.8 µm are required (7.31). The number of samples collected depends upon the specific circumstances of the study.

8.2 Maintain a log of all pertinent sampling information and sampling locations.

8.3 Sampling pumps and flow indicators shall be calibrated using a certified standard apparatus or assembly (see Practice D 3195 and 7.29).

8.4 *Record all calibration information (5).*

8.5 Perform a leak check of the sampling system at each sampling site by activating the pump (7.29) with the closed sampling cassette in line. Any air flow shows that a leak is present that must be eliminated before initiating the sampling operation.

8.6 Attach the sampling cassette to the sampling pump at the outlet side of the cassette with plastic tubing (7.35). The plastic tubing must be long enough in that the sample areas can be reached without interference from the sampling pump. Attach a clean, approximately 25.4 mm long piece of plastic tubing (6.35 mm internal diameter) directly to the inlet orifice. Use this piece of tubing as the sampling nozzle. Cut the sampling end of the tubing at a 45° angle as illustrated in Fig. 1. The exact design of the nozzle is not critical as long as some vacuum break is provided to avoid simply pushing the dust around on the surface with the nozzle rather than vacuuming it into the cassette. The internal diameter of the nozzle and flow rate of the pump may vary as long as the air velocity is 100 (±10) cm/s. This air velocity calculation is based on an internal sampling tube diameter of 6.35 mm at a flow rate of 2 L/min.

8.7 Measure and determine the sample area of interest. A

[6] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[7] Tygon is a registered trademark of the DuPont Co.

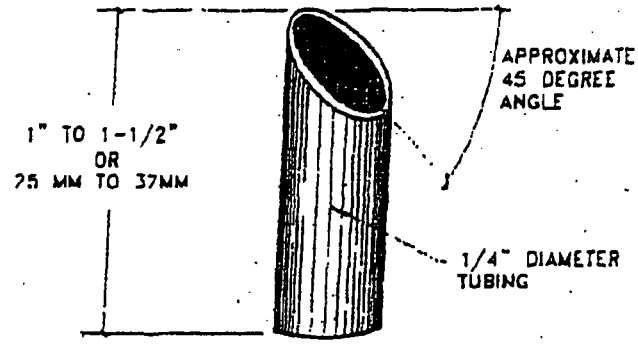

FIG. 1 Example of the Tubing Nozzle

sample area of 100 cm^2 is vacuumed until there is no visible dust or particulates matter remaining. Perform a minimum of two orthogonal passes on the surface within a minimum of 2 min of sampling time. Avoid scraping or abrading the surface being sampled. (Do not sample any debris or dust particles greater than 1 mm in diameter (see 4.2).) Smaller or larger areas can be sampled, if needed. For example, some surfaces of interest may have a smaller area than 100 cm^2. Less dusty surfaces may require vacuuming of larger areas. Unlike air samples, the overloading of the cassettes with dust will not be a problem. As defined in 3.2.5, only dust shall be collected for this analysis.

8.8 At the end of sample collection, invert the cassette so that the nozzle inlet faces up before shutting off the power to the pump. The nozzle is then sealed with a cassette end-plug and the cassette/nozzle taped or appropriately packaged to prevent separation of the nozzle and cassette assembly. A second option is the removal of the nozzle from the cassette, then plugging of the cassette and shipment of the nozzle (also plugged at both ends) sealed in a separate closeable plastic bag. A third option is placing the nozzle inside the cassette for shipment. The nozzle is always saved and rinsed because a significant percentage of the dust drawn from a lightly loaded surface may adhere to the inside walls of the tubing.

8.9 Check that all samples are clearly labeled, that all dust sampling information sheets are completed, and that all pertinent information has been enclosed, in accordance with laboratory quality control practices, before transfer of the samples to the laboratory. Include an unused cassette and nozzle as a field blank.

8.10 Wipe off the exterior surface of the cassettes with disposable wet towels (baby wipes) prior to packaging for shipment.

9. Sample Shipment

9.1 Ship dust samples to an analytical laboratory in a sealed container, but separate from any bulk or air samples. The cassettes must be tightly sealed and packed in a material free of fibers or dust to minimize the potential for contamination. Plastic "bubble pack" is probably the most appropriate material for this purpose.

10. Sample Preparation

10.1 Under a negative flow HEPA hood (7.5), carefully wet-wipe the exterior of the cassettes to remove any possible contamination before taking cassettes into a clean preparation area.

10.2 Perform sample preparation in a clean facility that has a separate work area from both the bulk and air sample preparation areas.

10.3 Initial specimen preparation shall take place in a clean HEPA filtered negative pressure hood to avoid any possible contamination of the laboratory or personnel, or both, by the potentially large number of asbestos structures in an asbestos-containing dust sample. Cleanliness of the preparation area hoods is measured by the cumulative process blank concentrations (see Section 11).

10.4 All sample preparation steps 10.4.1 through 10.4.6 shall take place in the dust preparation area inside a HEPA hood.

10.4.1 Remove the upper plug from the sample cassette and carefully introduce approximately 10 mL solution of a 50/50 mixture of particle-free water and reagent alcohol into the cassette using a plastic wash bottle (7.44). If the plugged nozzle was left attached to the cassette, then remove the plug and introduce the water/alcohol solution into the cassette through the tubing, and then remove the tubing, if it is visibly clean.

10.4.2 Replace the upper plug or the sample cap and lightly shake the dust suspension by hand for 3 s.

10.4.3 Remove the entire cap of the cassette and pour the suspension through a 1.0 by 1.0 mm opening screen (7.46) into a pre-cleaned 200 mL glass specimen bottle (7.9). All visible traces of the sample contained in the cassette shall be rinsed through the screen into the specimen bottle with a plastic wash bottle containing the 50/50 solution of particle-free water and alcohol. Repeat this procedure two additional times for a total of three washings. Next, rinse the nozzle two or three times through the screen into the specimen bottle with the 50/50 mixture of water and alcohol. Typically, the total amount of the 50/50 mixture used in the rinse is 50 to 75 mL. Discard the 1.0 by 1.0 mm screen and bring the volume of solution in the specimen bottle up to the 100 mL mark on the side of the bottle with particle-free water only.

10.4.4 Adjust the pH of the suspension to 3 to 4 using a 10.0 % solution of acetic acid. Use pH paper for testing. Filter the suspension within 24 h to avoid problems associated with bacterial and fungal growth.

10.4.5 Use either a disposable plastic filtration unit or a glass filtering unit (7.14) for filtration of aliquots of the suspension. The ability of an individual filtration unit to produce a uniform distribution may be tested by the filtration of a colored particulate suspension such as diluted India ink (suspension of carbon black).

10.4.5.1 If a disposable plastic filtration unit is used, then unwrap a new disposable plastic filter funnel unit (either 25 or 47 mm diameter) and remove the tape around the base of the funnel. Remove the funnel and discard the top filter supplied with the apparatus, retaining the coarse polypropylene support pad in place. Assemble the unit with the adapter and a properly sized neoprene stopper, and attach the funnel to the 1000 mL side-arm vacuum flask (7.15). Place a 5.0 µm pore size MCE (backing filter) on the support pad. Wet it with a few mL of particle-free water and place an MCE (7.16) or PC filter (≤0.22 µm pore size) (7.17) on top of the backing filter. Apply a vacuum (7.36), ensuring

that the filters are centered and pulled flat without air bubbles. Any irregularities on the filter surface requires the discard of that filter. After the filter has been seated properly, replace the funnel and reseal it with the tape. Return the flask to atmospheric pressure.

10.4.5.2 If a glass filtration unit is used, place a 5 µm pore size MCE (backing filter) on the glass frit surface. Wet the filter with particle-free water, and place an MCE or PC filter (≤0.22 µm pore size) on top of the backing filter. Apply a vacuum, ensuring that the filters are centered and pulled flat without air bubbles. Replace the filters if any irregularities are seen on the filter surface. Before filtration of each set of sample aliquots, prepare a blank filter by filtration of 50 mL of particle-free water. If aliquots of the same sample are filtered in order of increasing concentration, the glass filtration unit need not be washed between filtration. After completion of the filtration, do not allow the filtration funnel assembly to dry because contamination is then more difficult to remove. Wash any residual suspension from the filtration assembly by holding it under a flow of water, then rub the surface with a clean paper towel soaked in a detergent solution. Repeat the cleaning operation, and then rinse two times in particle-free water.

10.4.6 With the flask at atmospheric pressure, add 20 mL of particle-free water into the funnel. Cover the filter funnel with its plastic cover if the disposable filtering unit is used.

10.4.7 Briefly hand shake (3 s) the capped bottle with the sample suspension, then place it in a tabletop ultrasonic bath (7.12) and sonicate for 3.0 min. Maintain the water level in the sonicator at the same height as the solution in sample bottle. The ultrasonic bath shall be calibrated as described in 20.5. The ultrasonic bath must be operated at equilibrium temperature. After sonicating, return the sample bottle to the work surface of the HEPA hood. Preparation steps 10.4.8 through 10.4.14 shall be carried out in this hood.

10.4.8 Shake the suspension lightly by hand for 3 s, then let it rest for 2.0 min to allow large particles to settle to the bottom of the bottle or float to the surface.

10.4.9 Estimate the amount of liquid to be withdrawn to produce an adequate filter preparation. Experience has shown that a light staining of the filter surface will yield a suitable preparation for analysis. Filter at least 1.0 mL, but no more than half the total volume. If after examination in the TEM, the smallest volume measured (1.0 mL) (7.13) yields an overloaded sample, then perform additional serial dilutions of the suspension. If it is estimated that less than 1.0 mL of solution has to be filtered because of the density of the suspension, perform a serial dilution.

10.4.9.1 If serial dilutions are required, repeat step 10.4.8 before the serial dilution portion is taken. Do not re-sonicate the original solution or any serial dilutions. The recommended procedure for a serial dilution is to mix 10 mL of the sample solution with 90 mL of particle-free water in a clean sample bottle to obtain a 1:10 serial dilution. Follow good laboratory practices when performing dilutions.

10.4.10 Insert a new disposable pipette halfway into the sample suspension and withdraw a portion. Avoid pipetting any of the large floating or settled particles. Uncover the filter funnel and dispense the mixture from the pipette into the water in the funnel.

10.4.11 Apply vacuum to the flask and draw the mixture through the filter.

10.4.12 Discard the pipette.

10.4.13 Disassemble the filtering unit and carefully remove the sample filter with fine tweezers (7.11). Place the completed sample filter particle side up, into a precleaned, labeled, disposable, plastic petri dish (7.48) or other similar container.

10.4.14 In order to ensure that an optimally-loaded filter is obtained, it is recommended that filters be prepared from several different aliquots of the dust suspension. For this series of filters, it is recommended that the volume of each aliquot of the original suspension be a factor of five higher than the previous one. If the filters are prepared in order of increasing aliquot volume, all of the filters for one sample can be prepared using one plastic disposable filtration unit, or without cleaning of glass filtration equipment between individual filtration. Before withdrawal of each aliquot from the sample, shake the suspension without additional sonification and allow to rest for 2 min.

10.4.15 There are many practical methods for drying MCE filters. The following are two examples that can be used: (1) dry MCE filters for at least 12 h (over desiccant) in an airtight cabinet-type desiccator (7.21); (2) to shorten the drying time (if desired), remove a plug of the damp filter and attach it to a glass slide (7.19) as described in 12.1.2 and 12.1.3. Place the slide with a filter plug or filter plugs (up to eight plugs can be attached to one slide) on a bed of desiccant, in the desiccator for 1 h.

10.4.16 PC filters do not require lengthy drying before preparation, but shall be placed in a desiccator for at least 30 min before preparation.

10.5 Prepare TEM specimens from small sections of each dried filter using the appropriate direct transfer preparation method.

11. Blanks

11.1 Prepare sample blanks that include both a process blank (50 mL of particle-free water) for each set of samples analyzed and one unused filter from each new box of sample filters (MCE or PC) used in the laboratory. If glass filtering units are used, prepare and analyze a process blank each time the filtering unit is cleaned. Blanks will be considered contaminated, if after analysis, they are shown to contain more than 53 asbestos structures per square millimetre. This generally corresponds to three or four asbestos structures found in ten grid openings. The source of the contamination must be found before any further analysis can be performed. Reject samples that were processed along with the contaminated blanks and prepare new samples after the source of the contamination is found.

11.2 Prepare field blanks which are included with sample sets in the same manner as the samples, to test for contamination during the sampling, shipping, handling, and preparation steps of the method.

12. TEM Specimen Preparation of Mixed Cellulose Ester (MCE) Filters

NOTE 1—Use of either the acetone or the diamethylformamide-acetic acid method is acceptable.

12.1 *Acetone Fusing Method:*

12.1.1 Remove a section (a plug) from any quadrant of the sample and blank filters. Sections can be removed from the filters using a 7 mm cork borer (7.32). The cork borer must be wet wiped after each time a section is removed.

12.1.2 Place the filter section (particle side up) on a clean microscope slide. Affix the filter section to the slide with a gummed page reinforcement (7.43), or other suitable means. Label the slide with a glass scribing tool or permanent marker (7.10).

12.1.3 Prepare a fusing dish from a glass petri dish (7.37) and a metal screen bridge (7.38) with a pad of five to six ashless paper filters (7.42) and place in the bottom of the petri dish (4). Place the screen bridge on top of the pad and saturate the filter pads with acetone. Place the slide on top of the bridge in the petri dish and cover the dish. Wait approximately 5 min for the sample filter to fuse and clear.

12.2 *Dimethylformamide-Acetic Acid Method:*

12.2.1 Place a drop of clearing solution that consists of 35 % dimethylformamide (DMF), 15 % glacial acetic acid, and 50 % Type II water (v/v) on a clean microscope slide. Gauge the amount used so that the clearing solution just saturates the filter section.

12.2.2 Carefully lay the filter segment, sample surface upward, on top of the solution. Bring the filter and solution together at an angle of about 20° to help exclude air bubbles. Remove any excess clearing solution. Place the slide in an oven or on a hot plate, in a fume hood, at 65 to 70°C for 10 min.

12.3 Plasma etching of the collapsed filter is required.

12.3.1 The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher (7.27). Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the exact conditions that must be used. Insufficient etching will result in a failure to expose embedded fibers, and too much etching may result in the loss of particles from the filter surface. To determine the optimum time for ashing, place an unused 25 mm diameter MCE filter in the center of a glass microscope slide. Position the slide approximately in the center of the asher chamber. Close the chamber and evacuate to a pressure of approximately 40 Pa, while admitting oxygen to the chamber at a rate of 8 to 20 cm^3/min. Adjust the tuning of the system so that the intensity of the plasma is maximized. Determine the time required for complete oxidation of the filter. Adjust the system parameters to achieve complete oxidation of the filter in a period of approximately 15 min. For etching of collapsed filters, use these operating parameters for a period of 8 min. For additional information on calibration, see the *USEPA Asbestos-Containing Materials in Schools* (4) or *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis* (6) documents.

12.3.2 Place the glass slide containing the collapsed filters into the low-temperature plasma asher, and etch the filter.

12.4 Carbon coating of the collapsed and etched filters is required.

12.4.1 Carbon coating must be performed with a high-vacuum coating unit (7.4), capable of less than 10^{-4} torr (13 MPa) pressure. Units that are based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application and shall not be used. Carbon rods (7.40) used for evaporators shall be sharpened with a carbon rod sharpener to a neck of about 4 mm in length and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 100 to 120 mm from the surface of the microscope slide held in the rotating device.

12.4.2 Place the glass slide holding the filters on the rotation device, and evacuate the evaporator chamber to a vacuum of at least 13 MPa. Perform the evaporation in very short bursts, separated by 3 to 4 s to allow the electrodes to cool. An alternate method of evaporation is by using a slow continuous applied current. An experienced analyst can judge the thickness of the carbon film to be applied. Conduct tests on unused filters first. If the carbon film is too thin, large particles will be lost from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen.

12.4.2.1 If the coating is too thick, it will lead to a TEM image that is lacking in contrast, and the ability to obtain electron diffraction patterns will be compromised. The carbon film shall be as thin as possible and still remain intact on most of the grid openings of the TEM specimen.

12.5 *Preparation of the Jaffe Washer*—The precise design of the Jaffe washer is not considered important, so any one of the published designs may be used (7, 8). One such washer consists of a simple stainless steel bridge contained in a glass petri dish.

12.5.1 Place several pieces of lens tissue (7.41) on the stainless steel bridge. The pieces of lens tissue shall be large enough to completely drape over the bridge and into the solvent. In a fume hood, fill the petri dish with acetone (or DMF) until the height of the solvent is brought up to contact the underside of the metal bridge as illustrated in Fig. 2.

12.6 *Placing the Specimens into the Jaffe Washer:*

12.6.1 Place the TEM grids (7.39) shiny side up on a piece of lens tissue or filter paper so that individual grids can be easily picked up with tweezers.

12.6.2 Prepare three grids from each sample.

12.6.2.1 Using a curved scalpel blade (7.20), excise at least two square (3 mm by 3 mm) pieces of the carbon-coated MCE filter from the glass slide.

12.6.2.2 Place the square filter piece carbon-side up on top of a TEM specimen grid.

12.6.2.3 Place the whole assembly (filter/grid) on the saturated lens tissue in the Jaffe washer.

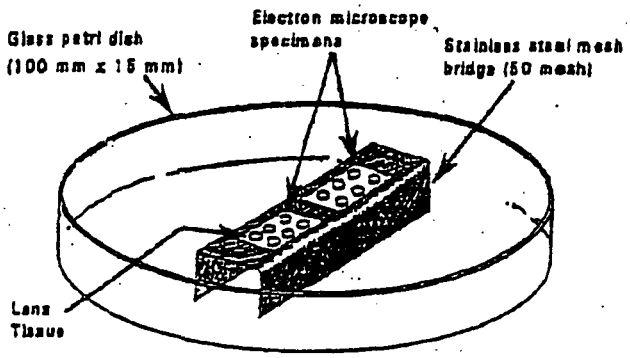

FIG. 2 Example of Design of Solvent Washer (Jaffe Washer)

12.6.2.4 Place the three TEM grid sample filter preparations on the same piece of lens tissue in the Jaffe washer.

12.6.2.5 Place the lid on the Jaffe washer and allow the system to stand for several hours.

12.7 Alternately, place the grids on a low level (petri dish filled to the ¼ mark) *DMF Jaffe washer for 60 min. Add* enough solution of equal parts DMF/acetone to fill the washer to the screen level. Remove the grids after 30 min if they have cleared, that is, all filter material has been removed from the carbon film, as determined by inspection in the TEM.

12.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean marked grid box.

13. TEM Specimen Preparation of Polycarbonate (PC) Filter

13.1 Cover the surface of a clean microscope slide with two strips of double-sided adhesive tape.

13.2 Cut a strip of filter paper slightly narrower than the width of the slide. Position the filter paper strip on the center of the length of the slide.

13.3 Using a clean, curved scalpel blade, cut a strip of the PC filter approximately 25 by 6 mm. Use a rocking motion of the scalpel blade to avoid tearing the filter. Place the PC strip particle side up on the slide perpendicular to the long axis of the slide. The ends of the PC strip must contact the double sided adhesive tape. Each slide can hold several PC strips. With a glass marker, label each PC strip with the individual sample number.

13.4 Carbon coat the PC filter strips as discussed in 12.4.2. PC filters do not require etching.

NOTE 2: Caution—Do not overheat the filter sections while carbon coating.

13.5 Prepare a Jaffe washer as described in 12.5, but fill the washer with chloroform or 1-methyl-2-pyrrolidone to the level of the screen.

13.6 Using a clean curved scalpel blade, excise three, 3-mm square filter pieces from each PC strip. Place the filter squares carbon side up on the shiny side of a TEM grid. Pick up the grid and filter section together and place them on the lens tissue in the Jaffe washer.

13.7 Place the lid on the Jaffe washer and rest the grids in place for at least 4 h. Best results are obtained with longer wicking times, up to 12 h.

13.8 Carefully remove the grids from the Jaffe washer, allowing the grids to dry before placing them in a clean, marked grid box.

14. Grid Opening Measurements

14.1 TEM grids must have a known grid opening area. Determine this area as follows:

14.2 Measure at least 20 grid openings in each of 20 random 75 to 100 µm (200-mesh) copper grids for a total of 400 grid openings for every 1000 grids used, by placing the 20 grids on a glass slide and examining them under the optical microscope. Use a calibrated graticule to measure the average length and width of the 20 openings from each of the individual grids. From the accumulated data, calculate the average grid opening area of the 400 openings.

14.3 Grid area measurements can also be made at the TEM at a calibrated screen magnification of between 15 000 and 20 000X. Typically measure one grid opening for each grid examined. Measure grid openings in both the x and y directions and calculate the area.

14.4 Pre-calibrated TEM grids are also acceptable for this test method.

15. TEM Method

15.1 Microscope settings: 80 to 120 kV, 15 000 to 20 000X screen magnification for analysis (7.2).

15.2 Analyze two grids for each sample. Analyze one-half of the sample area on one sample grid preparation and the remaining half on a second sample grid preparation.

15.3 *Determination of Specimen Suitability:*

15.3.1 Carefully load the TEM grid, carbon side facing up (in the TEM column) with the grid bars oriented parallel/perpendicular to the length of the specimen holder. Use a hand lens or loupe, if necessary. This procedure will line up the grid with the X and y translation directions of the microscope. Insert the specimen holder into the microscope.

15.3.2 Scan the entire grid at low magnification (250X to 1000X) to determine its suitability for high magnification analysis as specified in 15.3.3.

15.3.3 Grids are acceptable for analysis if the following conditions are met:

15.3.3.1 The fraction of grid openings covered by the replica section is at least 50 %.

15.3.3.2 Relative to that section of the grid covered by the carbon replica, the fraction of intact grid openings is greater than 50 %.

15.3.3.3 The fractional area of undissolved filter is less than 10 %.

15.3.3.4 The fraction of grid openings with overlapping or folded replica film is less than 50 %.

15.3.3.5 At least 20 grid openings, that have no overlapping or folded replica, are less than 5 % covered with holes and have less than 5 % opaque area due to incomplete filter dissolution.

15.4 *Determination of Grid Opening Suitability:*

15.4.1 If the grid meets acceptance criteria, choose a grid opening for analysis from various areas of the grid so that the entire grid is represented. Determine the suitability of each individual grid opening prior to the analysis.

15.4.2 The individual grid opening must have less than 5 % holes over its area.

15.4.3 Grid openings must be less than 25 % covered with particulate matter.

15.4.4 Grid openings must be uniformly loaded.

15.5 Observe and record the orientation of the grid at 80 to 150X, on a grid map record sheet along with the location of the grid openings that are examined for the analysis. If indexed grids are used, a grid map is not required, but the identifying coordinates of the grid square must be recorded.

16. Recording Data Rules

16.1 Record on the count sheet any continuous grouping of particles in which an asbestos fiber is detected. Classify asbestos structures as fibers, bundles, clusters, or matrices as defined in 5.2.

16.2 Use the criteria for fiber, bundle, cluster, and matrix identification, as described in the *USEPA Asbestos-Containing*

Materials in Schools document (4). Record, for each AHERA structure identified, the length and width measurements.

16.3 Record NSD (No Structures Detected) when no structures are detected in the grid opening.

16.4 Identify structures classified as chrysotile identified by either electron diffraction or X-ray analysis (7.3) and recorded on a count sheet. Verify at least one out of every ten chrysotile structures by X-ray analysis.

16.5 Structures classified as amphiboles by X-ray analysis and electron diffraction are recorded on the count sheet. For more information on identification, see Yamate, et al, (7) or Chatfield and Dillon (8).

16.6 Record a typical electron diffraction pattern for each type of asbestos observed for each group of samples (or a minimum of every five samples) analyzed. Record the micrograph number on the count sheet. Record at least one X-ray spectrum for each type of asbestos observed per sample. Attach the print-outs to the back of the count sheet. If the X-ray spectrum is stored, record the file and disk number on the count sheet.

16.7 *Counting Rules*:

16.7.1 At a screen magnification of between 15 000 and 20 000X evaluate the grids for the most concentrated sample loading; reject the sample if it is estimated to contain more than 50 asbestos structures per grid opening. Proceed to the next lower concentrated sample until a set of grids are obtained that have less than 30 asbestos structures per grid opening.

16.8 *Analytical Sensitivity*—An analytical sensitivity of approximately 1000 asbestos structures per square centimetre (calculated for the detection of a single asbestos structure) has been designed for this analysis. This sensitivity can be achieved by increasing the amount of liquid filtered, increasing the number of grid openings analyzed, or decreasing the size of the final filter. Occasionally, due to high particle loadings or high asbestos concentration, this analytical sensitivity cannot be practically achieved and stopping rules apply.

16.9 *Limit of Detection*—The limit of detection for this method is defined as, at a minimum, the counting of four asbestos structures during the TEM analysis. If less than four asbestos structures are counted during the analysis then the analytical result which will be reported will be less than the limit of detection and a "less than" sign (<) will appear before the number. All data shall be provided in the laboratory report.

16.10 *Stopping Rules*:

16.10.1 The analysis is stopped upon the completion of the grid square that achieves an analytical sensitivity of less than 1000 asbestos structures per square centimetre.

16.10.2 If an analytical sensitivity of 1000 asbestos structures per square centimetre cannot be achieved after analyzing ten grid openings then stop on grid opening No. 10 or the grid opening which contains the 100th asbestos structure, whichever comes first. A minimum of four grid squares shall be analyzed for each sample.

16.10.2.1 If the analysis is stopped because of the 100th structure rule, the entire grid square containing the 100th structure must be counted.

16.11 After analysis, remove the grids from the TEM, and replace them in the appropriate grid storage holder.

17. Sample Storage

17.1 The washed-out sample cassettes can be discarded after use.

17.2 Sample grids and unused filter sections (7.18) must be stored for a minimum of one year.

18. Reporting

18.1 Report the following information for each dust sample analyzed:

18.1.1 Concentration in structures/cm^2.

18.1.2 The analytical sensitivity.

18.1.3 Types of asbestos present.

18.1.4 Number of asbestos structures counted.

18.1.5 Effective filtration area.

18.1.6 Average size of the TEM grid openings that were counted.

18.1.7 Number of grid openings examined.

18.1.8 Sample dilution used.

18.1.9 Area of the surface sampled.

18.1.10 Listing of size data for each structure counted.

18.1.11 A copy of the TEM count sheet or a complete listing of the raw data. An example of a typical count sheet is shown in Appendix X1.

18.2 Determine the amount of asbestos in any accepted sample using the following formula:

$$\frac{EFA \times 100\ \text{mL} \times \#STR}{GO \times GOA \times V \times SPL} = \text{asbestos structures/cm}^2 \quad (1)$$

where:

$\#STR$ = number of asbestos structures counted,
EFA = effective filter area of the final sampling filter, mm^2,
GO = number of grid openings counted,
GOA = average grid opening area, mm^2,
SPL = surface area sampled, cm^2, and
V = volume of sample filtered in step 10.4.9, representing the actual volume taken from the original 100 mL suspension, mL.

19. Quality Control/Quality Assurance

19.1 In general, the laboratory's quality control checks are used to verify that a system is performing according to specifications regarding accuracy and consistency. In an analytical laboratory, spiked or known quantitative samples are normally used. However, due to the difficulties in preparing known quantitative asbestos samples, routine quality control testing focuses on re-analysis of samples (duplicate recounts).

19.1.1 Re-analyze samples at a rate of 1/10 of the sample sets (one out of every ten samples analyzed not including laboratory blanks). The re-analysis shall consist of a second sample preparation obtained from the final filter.

19.2 In addition, quality assurance programs must follow the criteria shown in the *USEPA Asbestos-Containing Materials in Schools* document (4) and in the *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis* document (6). These documents describe sample custody, sample preparation, blank checks for contamination, calibration, sample analysis, analyst qualifications, and technical facilities.

20. Calibrations

20.1 Perform calibrations of the instrumentation on a

regular basis, and retain these records in the laboratory, in accordance with the laboratory's quality assurance program.

20.2 Record calibrations in a log book along with dates of calibration and the attached backup documentation.

20.3 A calibration list for the instrument is as follows:

20.3.1 *TEM:*

20.3.1.1 Check the alignment and the systems operation. Refer to the TEM manufacturer's operational manual for detailed instructions.

20.3.1.2 Calibrate the camera length of the TEM in electron diffraction (ED) operating mode before ED patterns of unknown samples are observed. Camera length can be measured by using a carbon coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thick gold films will tend to mask weak diffraction spots from the fibrous particles. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings from thick films are unnecessary. Alternatively, a gold standard specimen can be used to obtain an average camera constant calculated for that particular instrument and can then be used for ED patterns of unknowns taken during the corresponding period.

20.3.1.3 Perform magnification calibration at the fluorescent screen. This calibration must be performed at the magnification used for structure counting. Calibration is performed with a grating replica (7.47) (for example, one containing at least 2160 lines/mm).

(*a*) Define a field of view on the fluorescent screen. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

(*b*) Frequency of calibration will depend on the service history of the particular microscope.

(*c*) Check the calibration after any maintenance of the microscope that involves adjustment of the power supply to the lens or the high voltage system or the mechanical disassembly of the electron optical column (apart from filament exchange).

(*d*) The analyst must ensure that the grating replica is placed at the same distance from the objective lens as the specimen.

(*e*) For instruments that incorporate a eucentric tilting specimen stage, all specimens and the grating replica must be placed at the eucentric position.

20.3.1.4 The smallest spot size of the TEM must be checked.

(*a*) At the crossover point, photograph the spot size at a screen magnification of 15 000 to 20 000X. An exposure time of 1 s is usually adequate.

(*b*) The measured spot size must be less than or equal to 250 nm.

20.4 *EDXA:*

20.4.1 The resolution and calibration of the EDXA must be verified.

20.4.1.1 Collect a standard EDXA Cu peak from the Cu grid.

20.4.1.2 Compare the X-ray energy versus channel number for the Cu peak and be certain that readings are within ±10 eV.

20.4.2 Collect a standard EDXA of crocidolite asbestos (NIST SRM 1866).

20.4.2.1 The elemental analysis of the crocidolite must resolve the Na peak.

20.4.3 Collect a standard EDXA of chrysotile asbestos.

20.4.3.1 The elemental analysis of chrysotile must resolve both Si and Mg on a single chrysotile fiber.

20.5 Ultrasonic bath calibration shall be performed as follows:

20.5.1 Fill the bath water to a level equal to the height of suspension in the glass sample container that will be used for the dust analysis. Operate the bath until the water reaches the equilibrium temperature.

20.5.2 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.3 Place the sample container in the water in the ultrasonic bath (with the power turned off). After 60 s, remove the glass container and record its temperature.

20.5.4 Place 100 mL of water (at approximately 20°C) in another 200-mL glass sample container, and record its temperature.

20.5.5 Place the second sample container into the water in the ultrasonic bath (with the power turned on). After 60 s, remove the glass container and record its temperature.

20.5.6 Calculate the rate of energy deposition into the sample container using the following formula:

$$R = 4.185 \times \sigma \times \rho \times \frac{(\theta_2 - \theta_1)}{t} \qquad (2)$$

where:

4.185 = Joules/cal,

R = energy deposition, watts/mL,

θ_1 = temperature rise with the ultrasonic bath not operating, °C,

θ_2 = temperature rise with the ultrasonic bath operating, °C,

t = time in seconds, 60 s (20.5.3 and 20.5.5),

σ = specific heat of the liquid in the glass sample container, 1.0 cal/g, and

ρ = density of the liquid in the glass sample container, 1.0 g/cm^3.

20.5.7 Adjust the operating conditions of the bath so that the rate of energy deposition is in the range of 0.08 to 0.12 MW/m^3, as defined by this procedure.

21. Precision and Bias

21.1 *Precision*—The precision of the procedure in this test method is being determined using round robin data from participating laboratories.

21.2 *Bias*—Since there is no accepted reference material suitable for determining the bias of the procedure in this test method, bias has not been determined (see Specification D 3670).

NOTE 3—Round robin data is under development and will be presented as a research report.

22. Keywords

22.1 asbestos; microvacuuming; settled dust; TEM

APPENDIX

(Nonmandatory Information)

X1. DUST SAMPLE ANALYSIS

X1.1 See Figs. X1.1 and X1.2 for the dust analysis worksheet and the TEM count sheet.

DUST SAMPLE ANALYSIS

Client:		Accelerating Voltage:	
Sample ID:		Indicated Mag:	KX
Job Number:		Screen Mag:	KX
Date Sample Analyzed:	- -	Microscope:	1 2 3 4 5
Number of Openings/Grids Counted:		Filter Type:	
Grid Accepted, 600X:	Yes No	Filter Size:	
Percent Loading:	%	Filter Pore Size (μm):	
Grid Box #1:		Grid Opening:	1) μm x μm
			2) μm x μm

Analyst: _____

Reviewer: _____ Counting Rules: AHERA LEVEL II

Calculation Data:

Effective Filter Area in mm^2:	(EFA)	
Number of Grid Openings Counted:	(GO)	
Avderage Grid Opening Area in mm^2:	(GOA)	
Volume of sample Filtered in ml:	(V)	
Surface area Sampled in cm^2:	(SPL)	
Number of Asbestos Structures Counted:*	(#STR)	

* If the number of asbestos structures counted is less than or equal to 4, enter 4 structures as the limit of detection here.

FORMULA FOR CALCULATION OF ASBESTOS STRUCTURES "DUST" PER CM^2:

$$\frac{EFAX \times 100 \times \#STR}{GO \times GOA \times VX SPL} = \text{(Asbestos Structures per } cm^2\text{)}$$

Results for Total Asbestos Structures: _____ (Structures per cm^2)

Results for Structures ≥ microns: _____ (Structures per cm^2)

Job Number: _____

Structure #	Grid # Square #	Type	Structure	Length Microns	Width Microns	Confirmation		
						Morph.	SAED	EDS

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(5) OSHA, *OSHA Technical Manual, OSHA Instruction CPL 2-2.DB*, Directorate of Technical Support, U.S. Department of Labor, Washington, DC 20210, Feb. 5, 1990, pp. 1–8 to 1–11.

(6) *NIST/NVLAP Program Handbook for Airborne Asbestos Analysis*, NISTIR, August 1989, pp. 89–4137.

(7) Yamate, G., Agarwall, S. C., and Gibbons, R. D., "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy," EPA Draft Report, Contract No. 68-02-3266, 1984.

(8) Chatfield, E. J., and Dillon, M. J., "Analytical Method for the Determination of Asbestos in Water," EPA No. 600/4-83-043, 1983.

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Appendix D
Logbook Pages

SOKKIA™

LEVEL
BOOK

TODD BURGESSER
LIBBY CSF
3/31/03 –

#100193

No. 8152-55

38 5/19/03-

Author: Todd Burgesser →

Governing Plan: CSF Soil Prep Plan 4/25/03 →

ISSI Libby 01 Rev 6 →

PPE - NA - →

Working on the Elastic D Base issues

Also - talk to Libby: T Keller about the negative air exposure assessment and filling out COC's

Packaged samples for shipment to Reservoir. 1 D sample ~~and~~ on shipment recieved - 3/15/03 COC #s D-125, D-126 and D-0128.

39

5/20/03

Author: Todd Burgesser →

Governing Plan: CSF Soil Prep 4/25/03 →

ISSI Libby 01 Rev 6 →

1500 COC entry to TB 5/20/03 into database from hardcopy COC →

Calibrated the drying oven 90.4°C →

Met wit K. Slaw on database issues

1600 left the lab for the office.

[signature] 5/20/03

5/21/03 Author Todd Burgesser.

Governing Plan: CSF Soil Prep Plan 4/25/03

ISSI - Libby oi Rev 6

PPE - Gloves, Respirator, safety glasses

Activities. Negative exposure assessment

0700 - Recieved pumps (personal) and will calibrate.

Pump 3142 calibrated with the buck calibrator # P 1378. Flow with the filter in line is 1.68 L/min.

Pump 2752 calibrated with the buck flowmeter # P 1378. Flow with filter is 1.58 L/min.

Pump 2752 attached to T. Clemans for grinding on the TWA sample at 0840. Tim Clemans has the cassette with cs# CS-11339.

Pump 3142 attached to K. Sloan for Sample Coordination activities and TWA sample cassette with CS# CS-11341.

Todd E. Burgesser 5/21/03

5/21/03 cont.

0845 - Start pump 3142 cs# 11341.

0915 - Calibrated pump 3146 with meter P1378. Flow rate with the cassette in line is 1.59 L/min. Cassette labelled with cs# CS-11342. Pump and cassette attached to K. Sloan TB 5/21/03 for the 30 minute excursion sample. Pump Started at 0948.

0950 - Pump ~~P1378~~ 3143 TB 5/21/03 calibrated with meter P1378. Flow rate with the cassette in line is 1.63 L/min. ~~Pum~~ TB 5/21/03 Cassette labelled with cs#, CS-11340. Pump and cassette attached to T. Clemans.

0957 - Pump 3143 started.

1020-1000 TB 5/21/03 - Pump 3146 attached to K Sloan faulted. Stopped sample and will submit the sample.

Todd E. Burgesser 5/21/03

5/21/03 - Post calibration on pump 3146 is 1.58 L/min. volume of air through cassette CS-11342 = 34.76 L. →

1027 ~~0907~~ TB 5/21/03 - Removed and turned off pump 3143 worn by T. Clemons. This pump was for the 30 min Excursion. pump post-calibrated to 1.63 L/min. Total flow through the cartridge was 48.9 L. on cartridge CS-11340.

1100 - Exit lab for office. To return this afternoon.

1330 - Back at lab. K Sloans pump (pump # 3142 had numerous faults. Recalibrate and total run time is 275 min.

1500 Removed Fran Clemons Pump ~~ZS~~ TB 2752 at 304 minutes. Calibrated the pump flow at 1.57 L/min.

Jadel E B 5/21/03

5/21/03 Cont. Cartridge # CS-11339 total flow - 477.28 Liters. This sample is the TWA sample.

1550 - Turned of K. Sloans pump. Collected CS-11348 from her pump pump calibrated at 1.67 L/min. TB

Hold samples until on 5/23/03 →

1540 - Changing the sample pumps → All day helped K Sloan with putting together COCs for shipment to Reservoir. Pulled and relabelled grinding blanks.

1645 - Exit Lab for the day.

Jadel E B 5/21/03

Author Todd Burgesser

5/22/03 At lab at 11:00 a.m. will not do the negative exposure assessment today. Spend afternoon organizing samples in Boxes. Review/QC entry of Batch 114 and 91 and FSDS # 000210.

Went through a box of miscellaneous WS-samples - Assisted T Clemans with the organization of the shelf in the back of the lab. Things to get for the lab. 2 fire ext. hook up shower & Eyewash. →

1630 exit lab for home. →

Todd Burgesser 5/22/03

5/23/03 - Author Todd Burgesser

Governing Plan CSF Soil Prep Plan 4/25/03

ISSF Libby 01 Rev 6 →

PPE: If in the lab, Safety glasses, gloves, Respirator. →

Activities: Negative exposure assessment.

0600 - Calibrate pumps for personal air sampling - Pump 2752 1.59 L/min. cassette on pump when calibrated. cassette # CS-11343, to go on T. Clemans when grampling. →

Calibrate pump # 3146. Flow rate is 1.73 L/minute with the cassette in line. Cassette # CS-11345

Flow meter used is the buck calibrator # P1378. Started pump 2752 on Tim Clemans @ 0720. Started pump 3146 on Kristen Sloan at 0741.

1000 Transfer of pump 3146 ~~2752~~ to T. Burgesser 5/23/03 so Kristen can get coffee and a Rice Krispy Treat. → TB 5/23/03

5/23/03 cont.

1022 - Switch pump 3146 back to Kristen Sloan from T. Burgesser.

1249 - Calibrate the pumps for the 30-minute excursion samples.

Pump 3143 calibrated at 1.59 L/min

pump 3143 had the cassette attached. Cassette # CS-11344

Pump 3142 calibrated at 1.66 L/min.

~~Sample~~ TB 5/23/03 Cassette # CS-11346 in line while callibrating.

1345 - pump 3142 placed on K. Sloan in breathing zone and turned on.

1415 Pump 3142 turned of and removed from Kristen Sloan.

1420 Pump 3142 post calibrated with the cassette in line. pump calibrated at 1.69 L/min. Put the samples for batch 54 back into the boxes ready for grind

Jadel E Burgesser 5/23/03

5/23/03 cont.

1443 - Attached and started pump 3143 to T. Clemans with cassette # CS-11344.

1516 - Disconnect and turn off pump 3143 worn by T. Clemans. Post calibration of pump

1517 3143 with cassette CS-11344 flow = 1.57 L/min.

1533 Pump 3146 removed and turned off from K. Sloan.

1534 Post calibration of 3146 with cassette CS-11345 1.69 L/min.

537 Removed and turned off pump 2752 with cassette CS-11343.

1539 Post calibration of pump 2752 with cassette in place is 1.59 L/m..

1541 - Place all cassettes in a ziplock bag for storage and shippened to a lab.

Jadel E B 5/23/03

[redacted] 5/23/03 cont.

1548 - Review/QC K. Sloan logbook

1600 - Exit lab for home →

5/23/03 [signature]

5/27/03 Author Todd Burgesser

Governing Plan: CSFSoil Prep Plan 4/25/03

ISSI Libby 01 Rev 6. →

PPE: Safty glasses, gloves, respirator. →

Activities Last day of the Negative air exposure assessment →

0700 Calibrate pump 2752 with cassette # CS-11347. Pump calibrated at 1.60 L/min.

0720 Pump 2752 attached to T Clemans and turned on. →

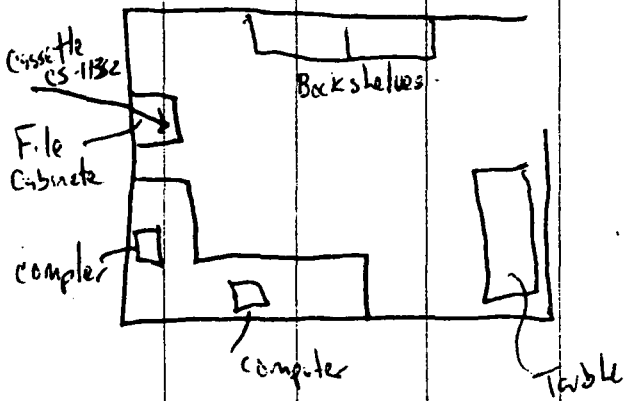
0725 Calibrated pump 3146 with cassette # CS-11349. Flow is 1.71 L/min.

0730 Calibrate pump HCA for the stationary air samples with manometer 003359. Calibrate with cassette CS-11351 at 8.41 L/minute. Pump HCA placed near grinding station and turned on at 0750. →

[signature] 5/27/03

5/27/03 cont.

0751 Calibrated pump 889-1055 with cassette CS-11352 in line. at 8.41 L/minute. Placed pump 889-1055 in the office Room at 0752 and turned it on. Pump 889-1055 placed on the file cabinet. See diagram below.

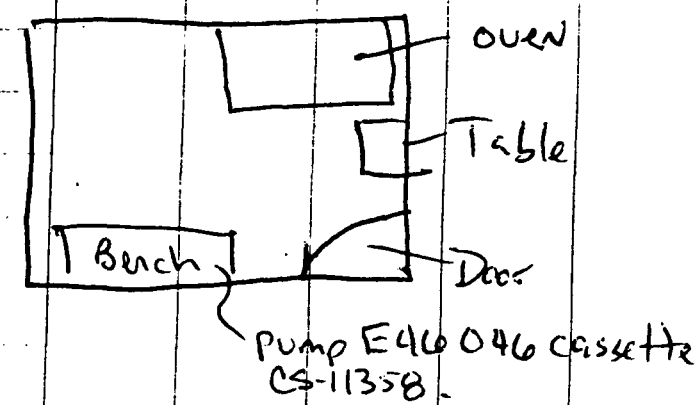


0803 Calibrate pump E46046 with manometer 0003359. and cassette # ~~CS-11353~~ T3 5/27/03 CS-11358. Pump E46046 calibrated at ~~8.39~~ 8.35 L/minute T3

[signature] 5/27/03

5/27/03 cont.

0808 - pump E46046 placed in over Room and turned on with cassette CS-11358.



0812: pump 0890 calibrated with cassette CS-11359 in line. pump calibrated at 8.41 L/min. pump 0890 place in sample recieving area and turned on at 0815 with cassette CS-11359.

0829. Pump 3146 with cassette CS-11349 put on K. Sloan and turned on

[signature] 5/27/03 T3

5/27/03 ~~0~~ 5/27/03 cont.

1240 - Calibration of pump 3143. with cassette in line CS-11348.

T35/27/03 ~~134~~ 1245 - Power outage and the stationary pumps were turned off.

1300 - Power returned to the lab and stationary pumps back on.

1305 - pump 3143 at 1.55 L/min - with cassette CS-11348

1306 pump 3143 placed on T. Clemans and turned on for 30 min excursion sample →

1307 - Calibration of pump 3142 with cassette CS-11350 in line, 1.78 L/min.

1311 Pump 3142 placed on K. Sloane and turned on. →

1339 - Removed pump from T. Clemans # 3143 with cassette CS-11348. Calibration of pump with flow meter P-1378 is 1.56 L/min. →

[signature] 5/27/03

5/27/03 cont.

1341 - Removed pump 3142 from K-Sloan with cassette 11350.

1343 - Post calibration of pump 3142 with meter P-1378 is 1.78 L/min.

1617 Remove pump 2752 with cassette CS-11347 from T. Clemans. Post calibration of pump 2752 is 1.58 L/min.

1621 - Stop pump 889-1055 in the office area with cassette # CS-11352. Post calibration of the pump with the cassette is 8.39 L/min.

1623 - Stop pump 0890 with cassette CS-11359. Post calibration is 7.66 L/min. Placed cassette in a baggy →

1626 Stopped pump E46046 in the Drying Room and post calibrated with cassette CS-11358. Post calibration measured 8.30 L/min.

[signature] 5/27/03.

54 5/27/03 cont.

1630 - Stopped pump HCA with cassette CS-11351. Post calibration of the pump with cassette is 8.29 L/min. TB 5/27/03 Cassette CS-11351 placed in a Baggie.

1637 - Removed pump 3146 from K. Sloan. Flow rate when calibrated was 1.680 L/min. Cassette # CS-11349 in line at time of post calibration.

1641 - Late entry. Calibration of the area using NIST Thermometer over calibrate at about 0630 am. 5/27/03.

1645 - Done for the day

[signature] 5/27/03

55

5/27/03 cont.

1650: Added 5 Samples ~~CS-11358~~ TB 5/27/03 CS-11358 and CS-11359 from the tracking sheet to be used as microvac samples.

[signature] 5/27/03

[redacted] Author [redacted] Burgesser [redacted]

Governing Plan CSF Soil Prep Plan 4/25/03 R

ISSI Lisby 01 Rev 6.

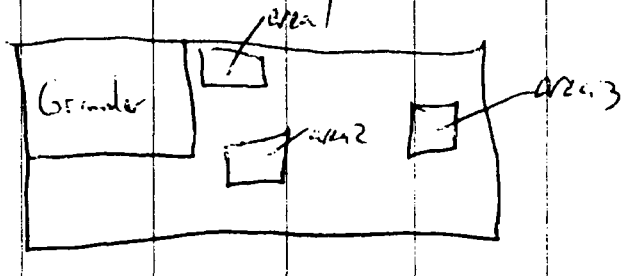
PPE - Gloves, Respirator, safety glasses

Activities: collection of microvac dust samples from office, Drying Room Lab and sample receiving room.

1007 calibration of pump 3146 with flow meter P1378. Flow rate = 2.15 L/min, with cassette CS-11360

1010 Attached tube to cassette CS-11360.

1011 Vaccume 3 - 100 cm² areas in the fume hood in three areas as shown.



1017 - Stop Pump

[illegible] 4/28/03

1018 Post calibrate pump.

3146 with meter P1378

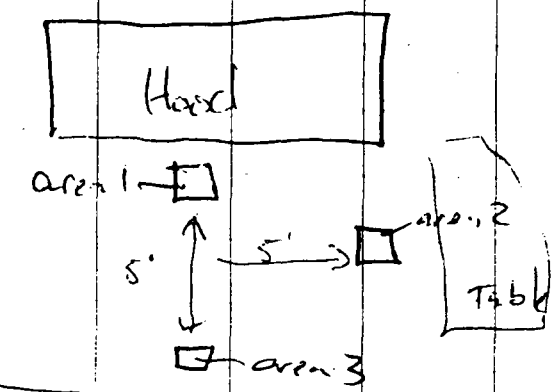
flow rate 2.15 cm³ 5/28/03 L/min.

Vaccumed each 100 cm² area for 2 minutes using two rulers as templates.

TG 5/28/03

1020 - calibrate pump 3146 with the meter P1378. Flow rate is 2.15 L/m with cassette CS-11361 in line.

1023 With cassette CS-11361 will vac prep lab area in front of hood as shown below:



[illegible] 5/28/03

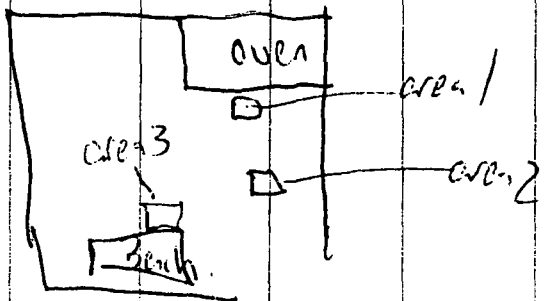
5/28/03 cont.

1024 - Turn on pump 3146 and vacuume lab area with cassette CS-11361. →

1030 - Turned of pump 3146 with cassette CS-11361. Post calibration of the pump is 2.15 l/min with meter P-1378.

1034 Callibrate pump 3146 with cassette # CS-11362 = 2.14

1041 - start pump CS-11362 cassette pump # 3146, in soil drying room. Vacuume areas as shown using Rulers as template.

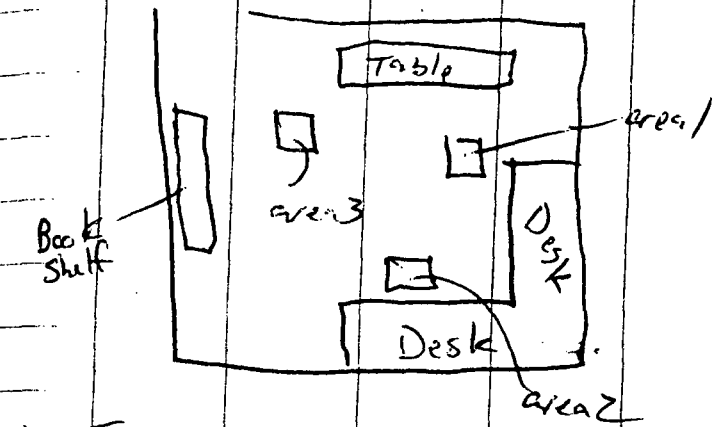


[signature] 5/28/03

5/28/03 cont.

1047 - Turn of pump with cassette CS-11362. Post calibration of pump with meter P-1378 = 2.14 L/min.

1050 Calibrate pump 3146 with cassette CS-11363 in line. Flow = 2.15 l/min. will vacuum the office room in 3 places (100 cm² each) as shown.



1052 - Turn on pump 3146 with cassette CS-11363 and vacuum the office area. →

1058 - Turn off pump with CS 11363 in line. →

[signature] 5/28/03

5/28/03 cont.

Post calibrate pump 3146 with cassette CS-11363 in line. flow rate is 2.15 l/min. →

1100: fill out COCs.

1130: Deliver all samples for the negative exposure to Reservoir.

1600 Created CS-11365 for grinding Blank. Enter Information into Elastic. →

1610 QC of FSDS form and of the log calibration logs. all ok. →

TB 5/28/03

5/29/03 - Author: Todd Burgessa

Governing Plan CSF Soil Prep Plan 4/25/03 ISSI 01 Rev 6 →

PPE: Gloves, safety glasses and Respirator when in the lab.

Activities: Pulling samples from batch group 5/28/03. A number of CS samples have already been sent for analysis. →

Reformatted the spreadsheet that Terry Keller gave me and and sent it back to all who were attached. Mark Raney (Volpe) wants to see the suffix IDs on the sheet. →

Pulled samples from Phase 1 Boxes for the 5/28/03 Batch of sample. Checked

1535 The Calibration Logs and Batch forms - Checked the GB for today →

TB 5/29/03